Agilent 6890N Gas Chromatograph

User Information

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Analytical Sequences

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30. Making SWAGELOK Connections

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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 CISPR 11. 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 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.

Sound Emission Certification for Federal Republic of Germany

Sound Pressure

Sound pressure Lp < 65 dB(A) according to DIN-EN 27779.

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

Schalldruckpegel

Schalldruckpegel LP < 65 dB(A) nach DIN-EN 27779.

Bei Betrieb des 6890 mit Cryo Ventil Option treten beim Oeffnen des Ventils kurzfristig Impulse bis zu einem Schalldruckpegel LP von ca.74.6 dB(A) auf.

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1 General Information

Control tables

Using control tables

Some specifics

- Gas control
- Columns
- Signals
- Automation
- Methods and sequences
- Valves

Strategy

Maintenance information

- Fuses and batteries
- Maintenance schedule

General warnings

- Many internal parts of the GC carry dangerous voltages
- Electrostatic discharge is a threat to GC electronics
- Many parts are dangerously hot

Shutting down the GC

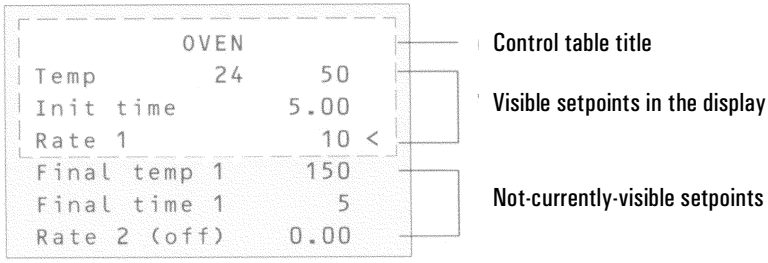
- For less than one week
- For more than one week

The 6890 Series Gas Chromatograph

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

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.

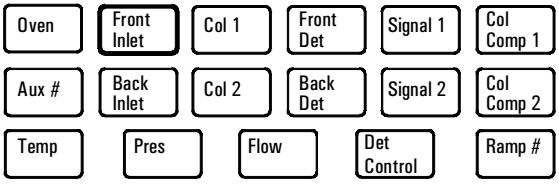
Using control tables

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 shown in Figure 1.

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 controlers, 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!

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 μ-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

The display

The status board

The keyboard

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

Function keys

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

[Info]

[Status]
The Ready/Not Ready status table
The setpoint status table
Procedure: Configuring the set-point status table

Miscellaneous keys
[Time]
Procedure: Setting time and date
Procedure: Using the stopwatch
Procedure: Setting up [Post Run]
[Run Log]
[Options]
[Config]

Modifier keys
[Mode/Type]
[Clear]
[Delete]
[.]
[–]

Storage and automation

Default parameters
Procedure: Loading the default parameters

The Keyboard and Display

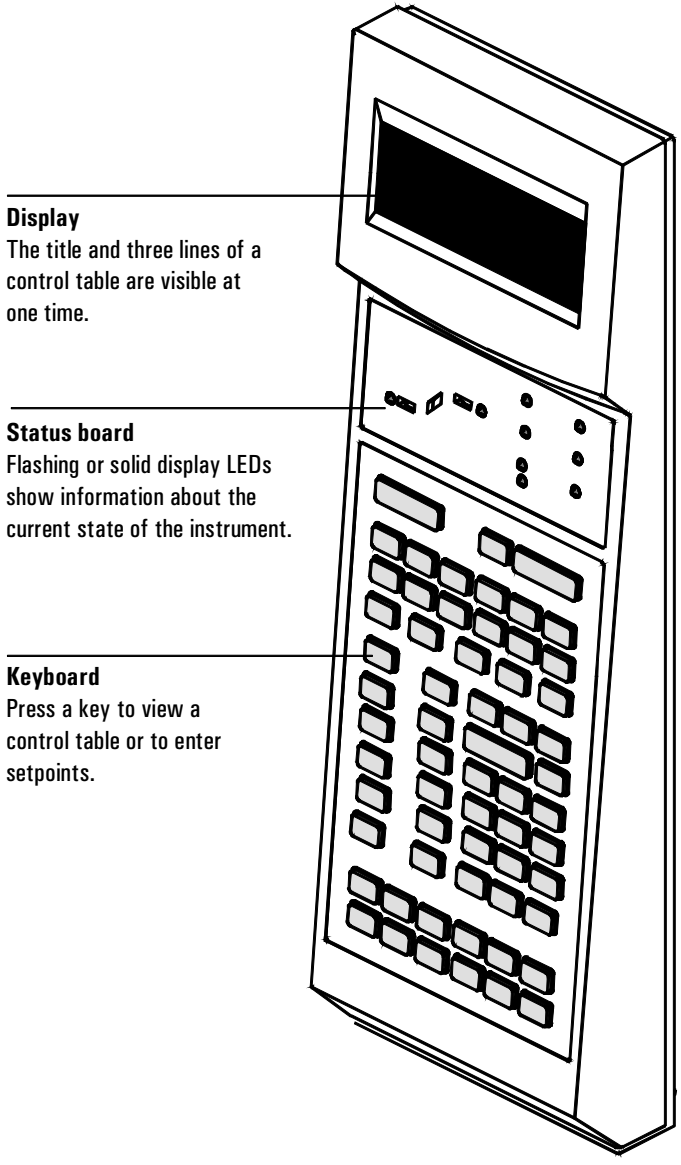


Figure 2 The GC controls

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 (▲,▼)

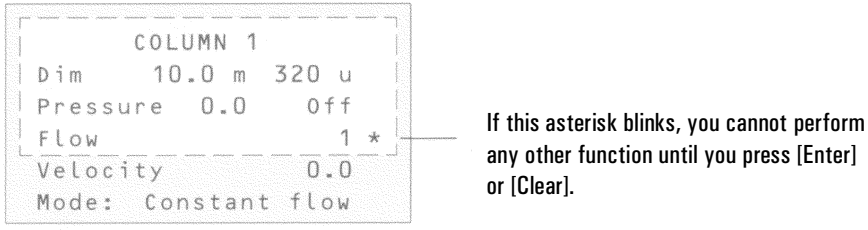
Move the control table up or down in the display window.

The cursor (<)

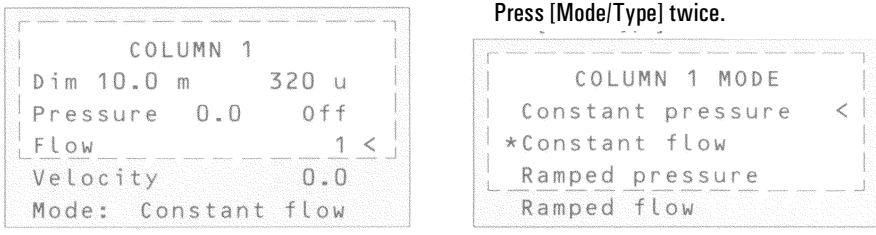
Points to the line that is in editing position. Changes that you make using the keyboard apply to the "cursored" line.

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.



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



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.

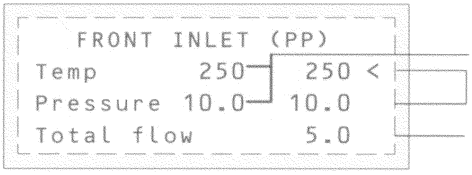
Blinking setpoint

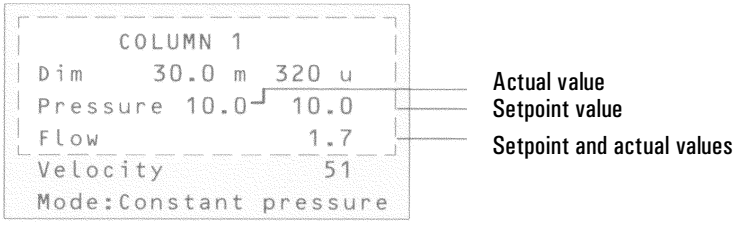
If a gas flow, multiposition valve, or the oven is shut down by the system, `Off` will blink at the appropriate line of the control table. This helps you identify where the problem occurred.

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.



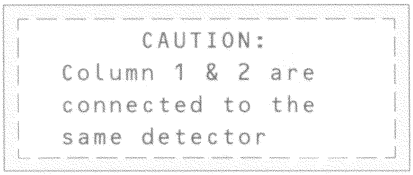


Messages

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:

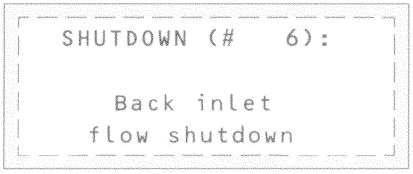
- The setpoint you've entered is out of the allowable range.
- You do not have the hardware on your instrument to support the operation you have requested.

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 "Warning".

Popup message:



Press [Clear] to remove the message.

The status board

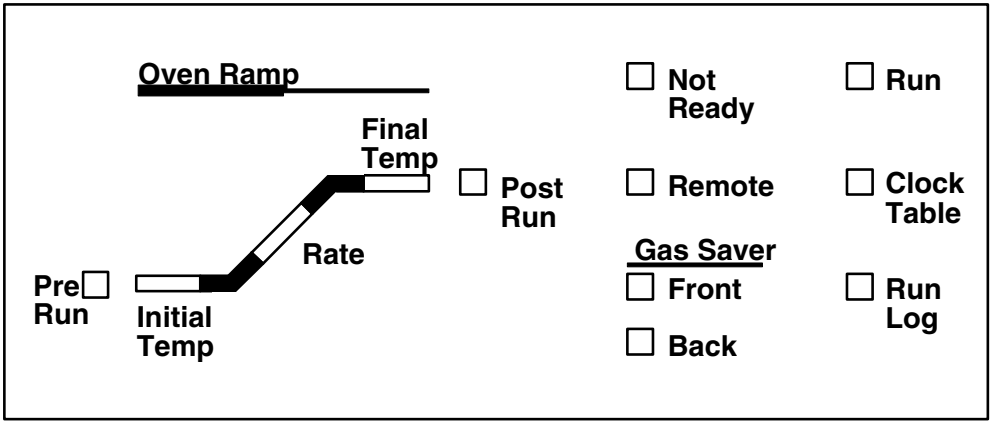


Figure 3 The status board

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 page 285 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

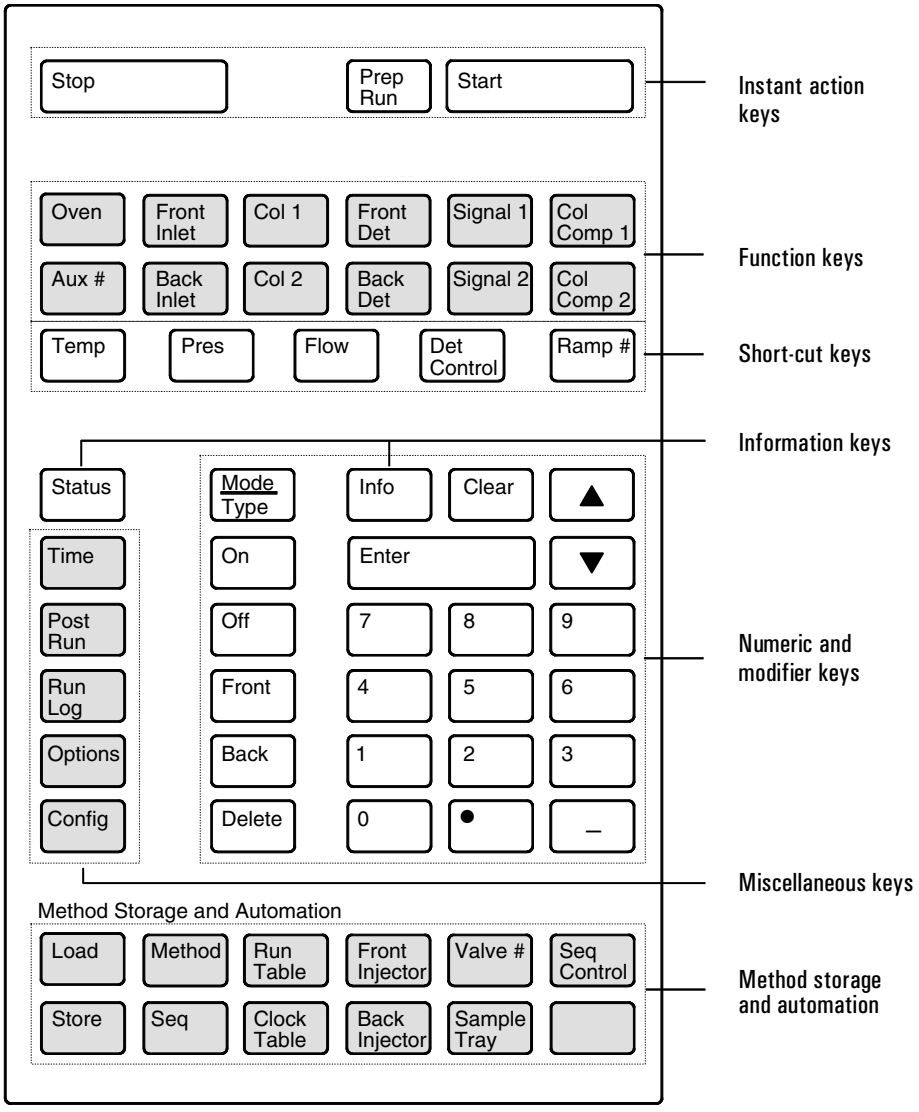


Figure 4 The keyboard

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 a 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 "[Config]" on page 56.

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.	See "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.	See "Valve Control".
[Aux #] [3], [Aux #] [4], and [Aux #] [5]	Provide auxiliary pneumatics to an inlet, detector, or other device. Can do pressure programming.	See "Flow and Pressure Control" and "Valve Control".
[Front Inlet] and [Back Inlet]	Control inlet operating parameters.	See "The Split/Splitless Inlet", "The Purged Packed Inlet", "The Cool On-Column Inlet", "The Programmable Temperature Vaporization Inlet", "The Volatiles Interface", "NonEPC Inlets", "The Pneumatics Control Module".
[Col 1] and [Col 2]	Control column pressure, flow, or velocity. Can set pressure or flow ramps.	See "Flow and Pressure Control", "The Split/Splitless Inlet", "The Purged Packed Inlet", "The Cool On-Column Inlet", "The Programmable Temperature Vaporization Inlet", "The Volatiles Interface", "NonEPC Inlets", "The Pneumatics Control Module", "The Flame Ionization Detector", "The Thermal Conductivity Detector", "The Nitrogen-Phosphorus Detector", "The Micro-Cell Electron Capture Detector", "The Flame Photometric Detector", .
[Front Det] and [Back Det]	Control detector operating parameters.	See "The Flame Ionization Detector", "The Thermal Conductivity Detector", "The Nitrogen-Phosphorus Detector", "The Micro-Cell Electron Capture Detector", "The Flame Photometric Detector".
[Signal 1] and [Signal 2]	Assign a signal, usually to the front or back detector.	See "Signal Handling".
[Col Comp 1] and [Col Comp 2]	Create a column compensation profile.	See "Signal Handling".

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

Quickly access a setpoint from within a table.

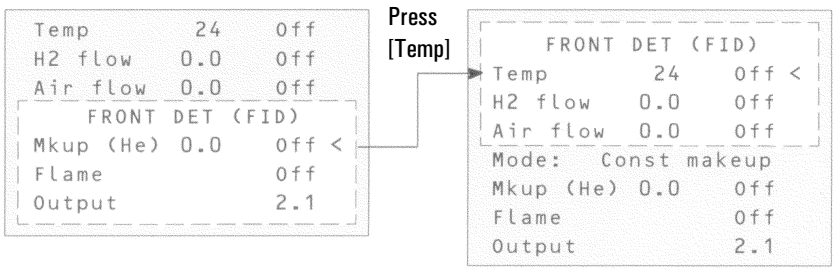
[Temp], [Pres], and [Flow]

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)`



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

```
          OVEN
Temp        24      50
Init time           5.00
Rate 1                10 <
Final temp 1         150
Final time 1           5
Rate 2 (off)        0.00
```

Press [Pres]

[Front inlet] table opens

```
   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]

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]

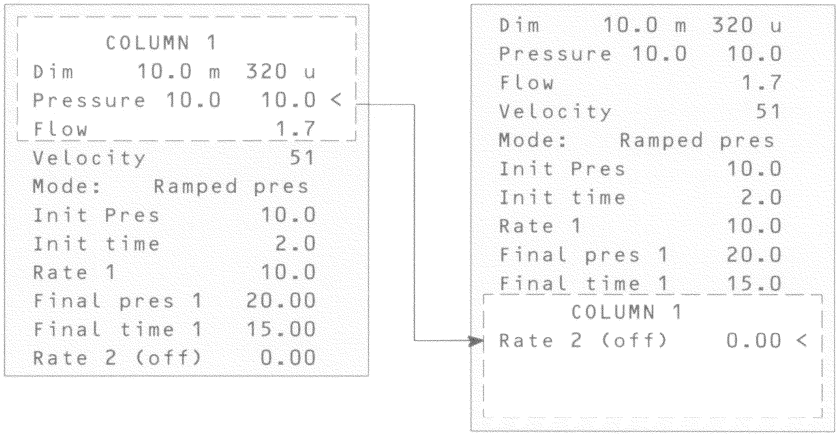
```
          OVEN

Rate 1 (off)     0.00 <
```

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]

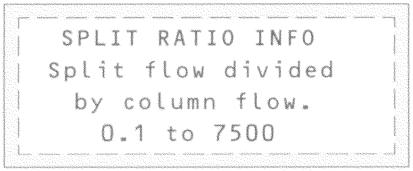
This is a context-sensitive help that provides information about an active parameter (line with the cursor).

These info messages may be in several different forms,

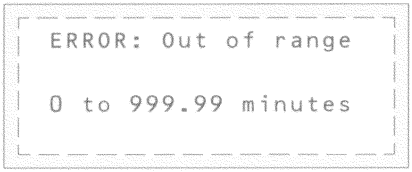
- Definitions
- Setpoint ranges
- Actions to perform

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

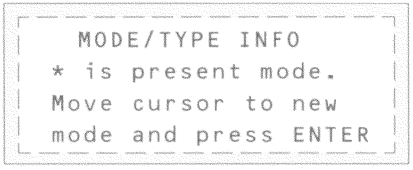
Definition:



Setpoint ranges:



Perform an action:



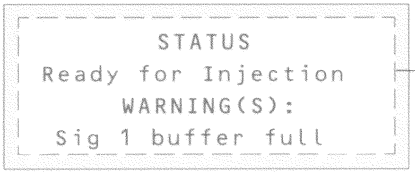
[Status]

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

The Ready/Not Ready status table

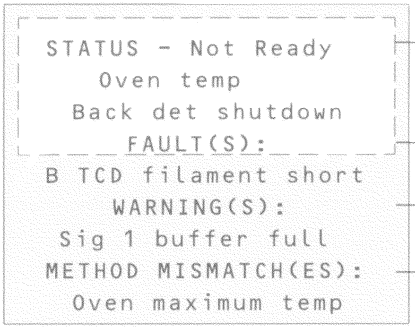
This table lists parameters that are *Not Ready* or gives you a *Ready for Injection* display. If there are any *faults*, *warnings*, or *method mismatches* present, they are displayed here. See page 202 and page 242 for detailed information about the not ready, fault, and warning status displays. The method mismatch displays are discussed on page 202.

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.

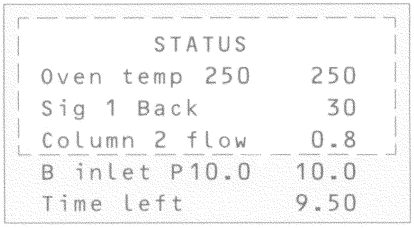
Fault—a hardware problem requiring user intervention.

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.



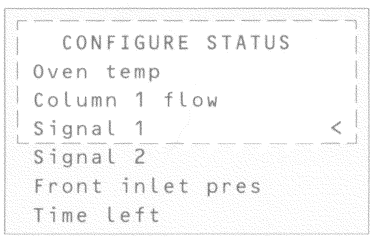
Procedure: Configuring the setpoint status table

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.

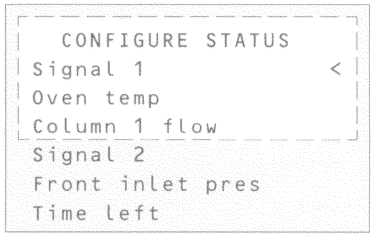
1. Press [Config] [Status].
2. Scroll to the setpoint that should appear first and press [Enter]. This setpoint will now appear at the top of the list.
3. Scroll to the setpoint that should appear second and press [Enter]. This setpoint will now be the second item on the list.
4. And so on, until the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

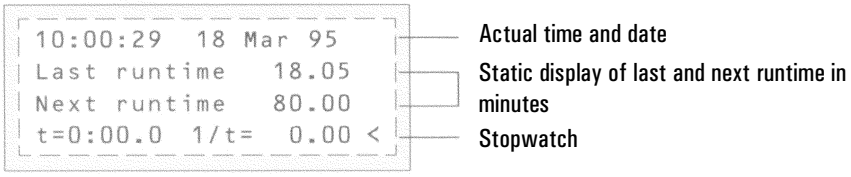


Miscellaneous keys

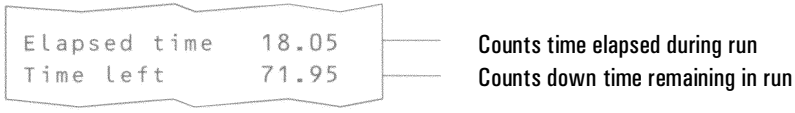
[Time]

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.

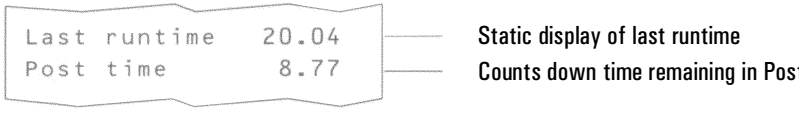
Time display between runs



Time display during a run

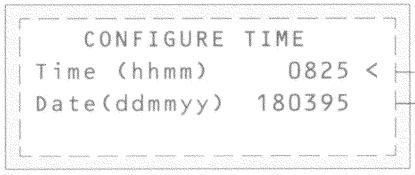


Time display during Post Run



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.

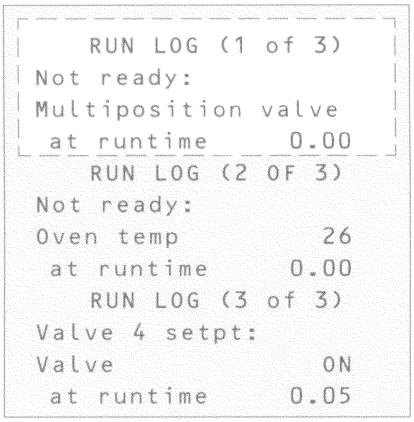
If you press [Time] while in a Post Run, you can view the amount of time remaining.

[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]



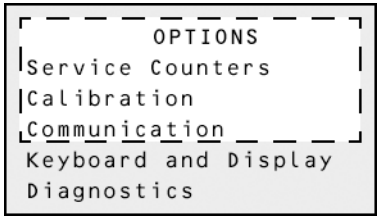
The Run Log LED is lit if there are any entries in the run log for the run in progress. The run log is cleared at the start of a new run.

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 on "Installation".

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.

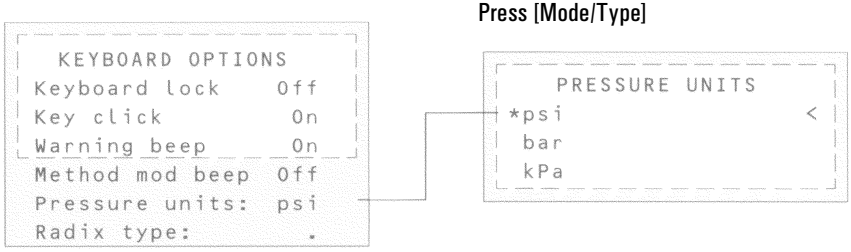
Press [Mode/Type] to change the pressure units and radix type.

- `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

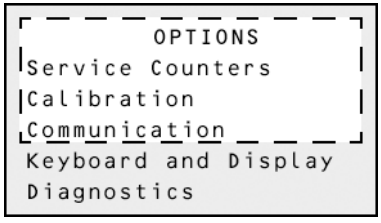
- `Radix type`—determines the numeric separator type—1.00 *or* 1,00



Service Counters

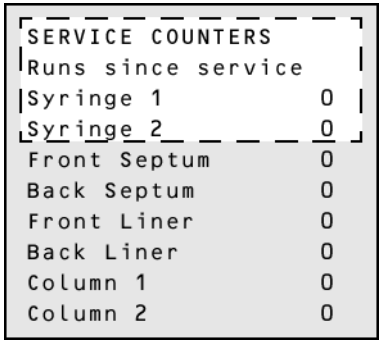
Tracks syringe, septum, and liner usage by counting each injection (regardless of Front/Back or INJ type).

1. At keyboard, press [Options].



2. From the open control table, select Service Counters. Press [Enter].

3. Scroll to desired counter. Press [Clear] to reset counter to 0.

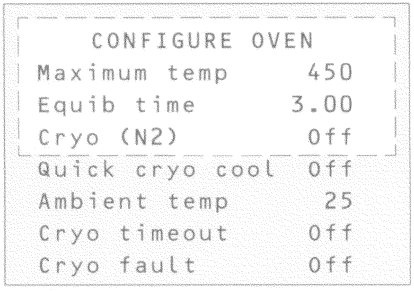


[Config]

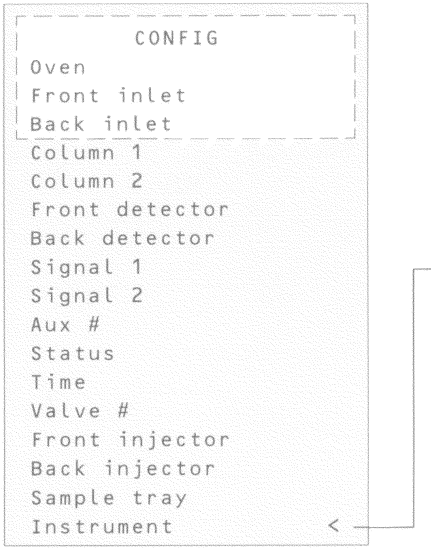
The [Config] key sets up configurations for instrument control. The column mode and dimensions, inlet, and makeup gas type configurations are critical to proper operation of EPC.

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

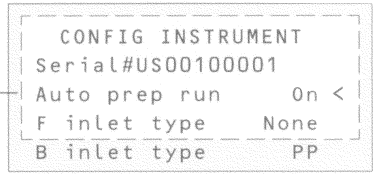
Press [Config] [Oven]



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

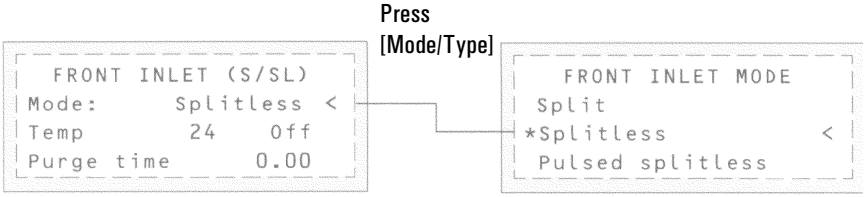
Modifier keys extend the functions of some setpoint control keys.

[Mode/Type]

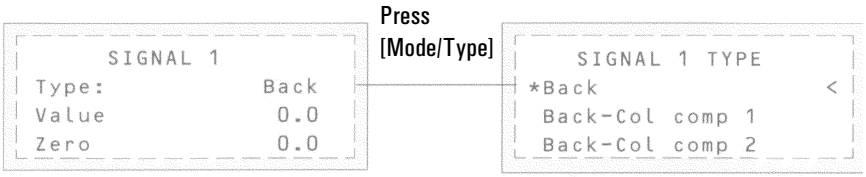
Use this key to access a list of possible modes or types associated with nonnumeric setpoints. To change mode or type, scroll to the desired line and press [Enter]. An asterisk (*) marks the present mode or type.

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

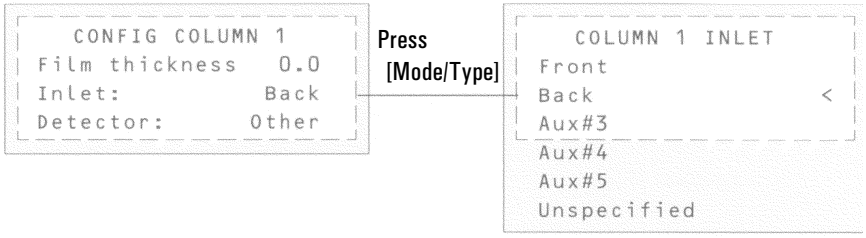
Mode:



Type:



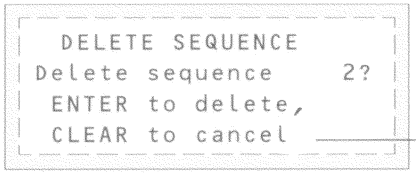
Examples of instances when the words “Mode” or “Type” do not appear. When in doubt, press [Info] to find out if [Mode/Type] is to be used.



[Clear]

The [Clear] key is used to:

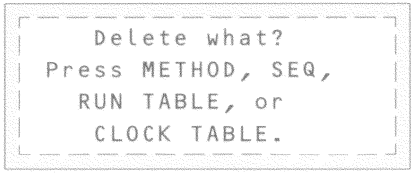
- Clear mis-entered setpoints in a control table *before* pressing [Enter] (when the * is still flashing).
- Back out of Mode/Type select before pressing [Enter].
- Return to upper level in nested control tables (config, option).
- Clear the stopwatch to zero.
- Clear info message and return to previous 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.

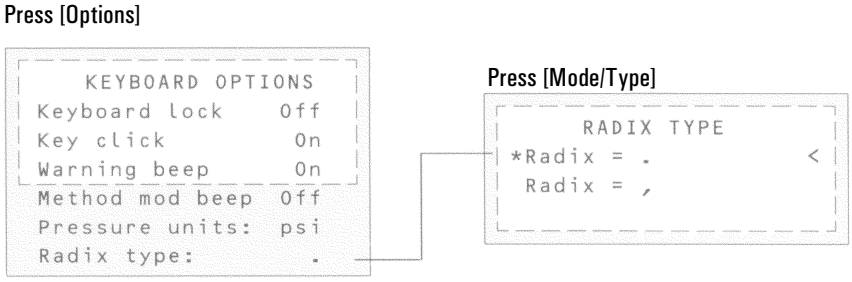
Press [Delete]



[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 nine stored methods and five stored sequences.	"Analytical Methods" "Analytical Sequences"
[Store]	Store up to nine methods and five sequences. Stored methods and sequences are labeled and dated.	"Analytical Methods" "Analytical Sequences"
[Method]	Review a table of stored methods. You can load, store, delete, or set default method.	"Analytical Methods"
[Seq]	Review a table of stored sequences. The [Seq] key toggles between the stored sequences control table and sequence definition control table.	"Analytical Sequences"
[Run Table]	View a table of events and the run time at which they occur.	"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.	"Instrument Automation"
[Front Injector] or [Back Injector]	Edit injector control parameters such as injection volumes, sample and solvent washes, etc.	"The Automatic Sampler"
[Valve#]	Turn GSV and selection valves 1 to 8 on or off. Sets multiposition valve position.	"Valve Control"
[Sample Tray]	Display the tray status.	"The Automatic Sampler"
[Seq Control]	Start, stop, pause or resume a sequence, and view sequence status.	"Analytical Sequences"

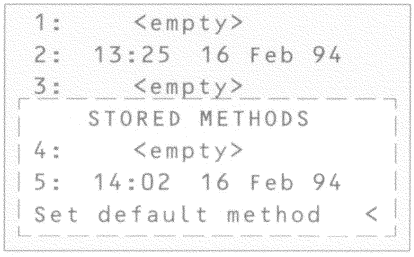
Default parameters

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.

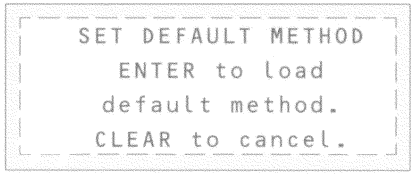
At some time, you may find it desirable to reload the default parameters. Doing this erases all current parameters except any methods you have stored and replaces them with the default set.

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

Hydrogen shutdown

Column shutdown

Turning gas flows on and off
EPC-controlled streams
NonEPC-controlled streams

Electronic Pneumatic Control (EPC)

Interpreting flow and pressure readings

Configuration

Columns and inlets

Configure the column
Procedure: Configuring a capillary column
Additional notes on column configuration

Configure the carrier gas
Procedure: Configuring the carrier gas

Select a column mode
The flow modes
The pressure modes
Procedure: Selecting a column mode

Enter the initial flow or pressure or average linear velocity
Procedure: Setting initial flow or pressure or average linear velocity

Enter a flow or pressure program (optional)
Procedure: Programming column pressure or flow

Enter the rest of the inlet parameters
Procedure: Setting the rest of the inlet parameters

Detectors
Gas configuration
Makeup gas

Auxiliary channels
Procedure: Changing an auxiliary channel frit

Maintaining EPC calibration
Flow sensors
Pressure sensors
Zero conditions
Procedure: Zeroing flow and pressure sensors

NonEPC control

Inlets
Septum purge

Measuring flow rates

Measuring flow rates with a bubble meter

Where to measure flows

Adapters for measuring flow rates

Procedure: Measuring gas flows with a bubble meter

Interpreting flow meter measurements

Flow and pressure problems

A gas does not reach the setpoint pressure or flow

A gas exceeds the setpoint pressure or flow

The inlet pressure or flow fluctuates

The measured flow is not equal to the displayed flow

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

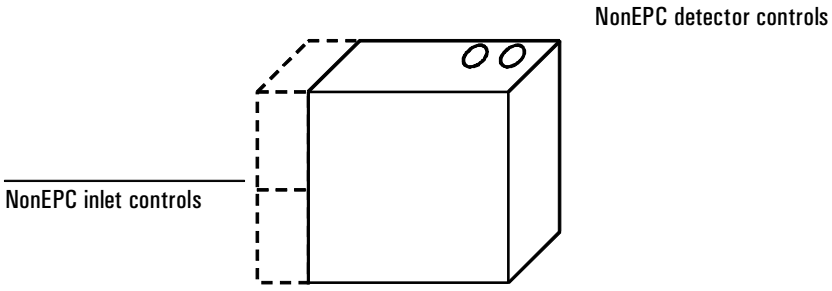


Figure 5 Control location
The pneumatics module (dashed lines) is present if a nonEPC inlet is installed.

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.

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 "Introduction to Inlets" and "Using Detectors".

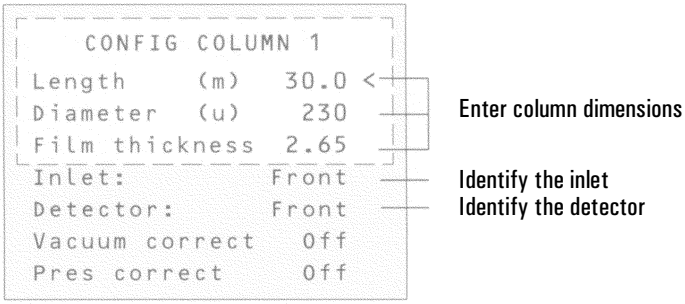
Configure the column

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 *"Introduction to Inlets"* and *"Using Detectors"* 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.

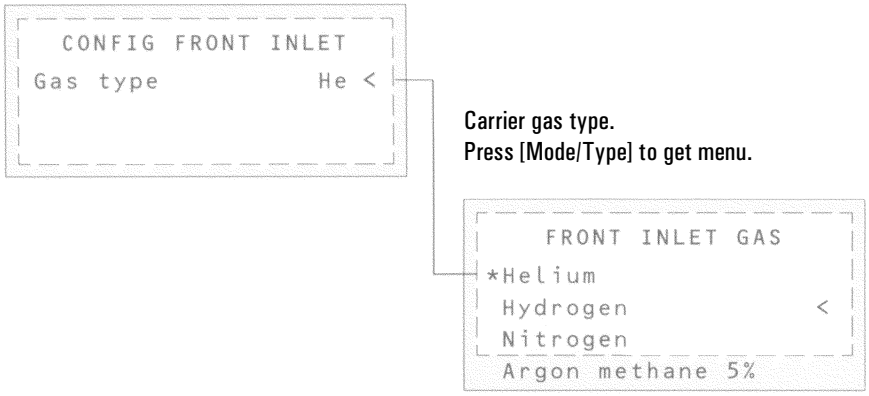
  It is possible, and sometimes appropriate, to configure both installed columns to the same inlet.
- Some pneumatic setpoints change with oven temperature because of changes in column resistance and in gas viscosity. This may confuse users who observe pneumatics setpoints changing when their oven temperature 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 column control table—packed or undefined capillary columns"* 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 67 and 98.

- **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

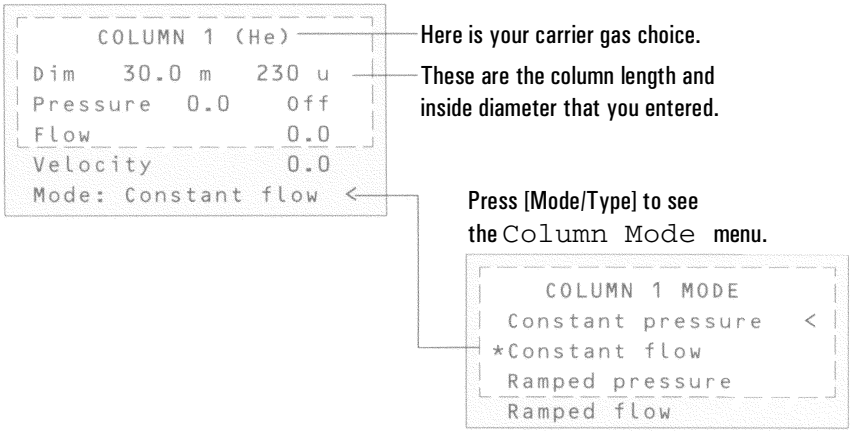
Pressures are gauge pressures—the difference between the absolute pressure and the local atmospheric pressure. Because most detectors present little resistance to the column flow, the gauge pressure at the column head is usually the same as the pressure difference between column inlet and exit. The mass 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

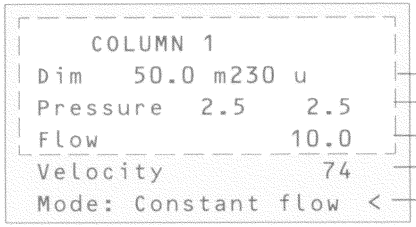
		Carrier gas flow rate	
Column type	**Column size**	**Hydrogen**	**Helium**
Packed	1/8-inch		30
	1/4-inch		60
Capillary	50 µm id	0.5	0.4
	100 µm id	1.0	0.8
	200 µm id	2.0	1.6
	250 µm id	2.5	2.0
	320 µm id	3.2	2.6
	530 µm id	5.3	4.2

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

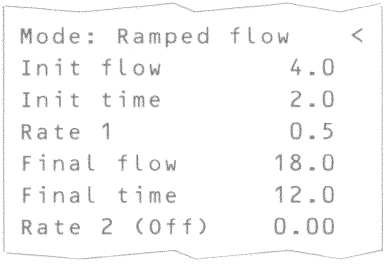
For capillary columns, flow rates are proportional to column diameter and are 20% lower for helium than for hydrogen.

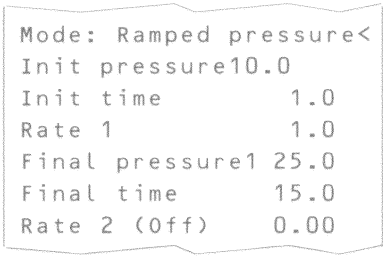
Procedure: Setting initial flow or pressure or average linear velocity

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`.

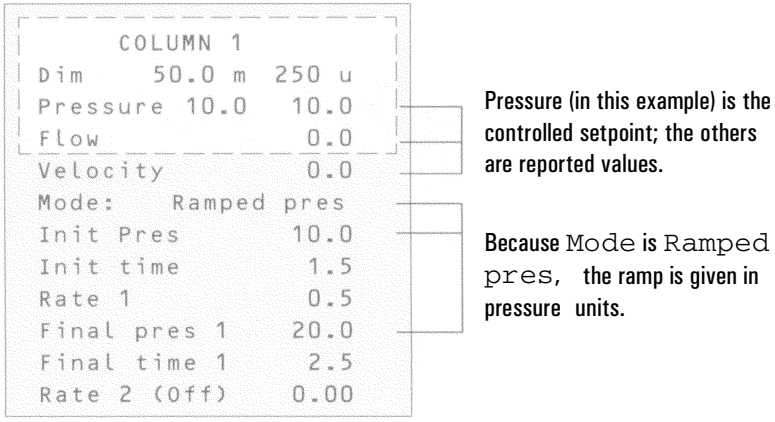
The program ends when it reaches a `Rate` that is set to 0 (off).

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.

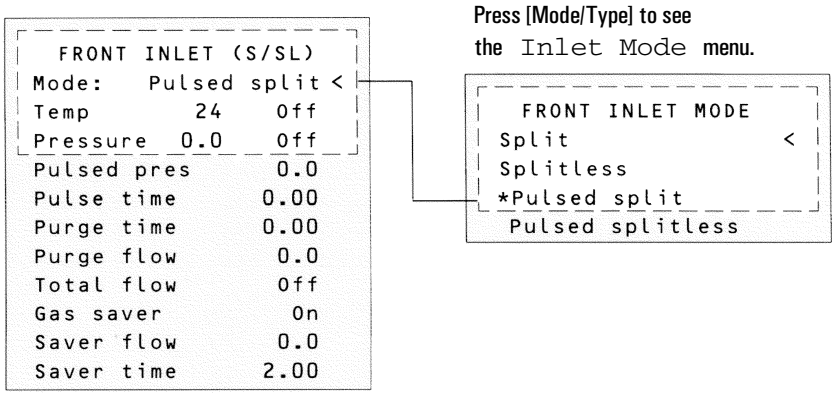
The split/splitless inlet has a gas saver feature. This reduces the flow of carrier into the inlet and out the split vent after the injection is complete. It does not alter the flow through the column.

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 *"Introduction to Inlets"*.

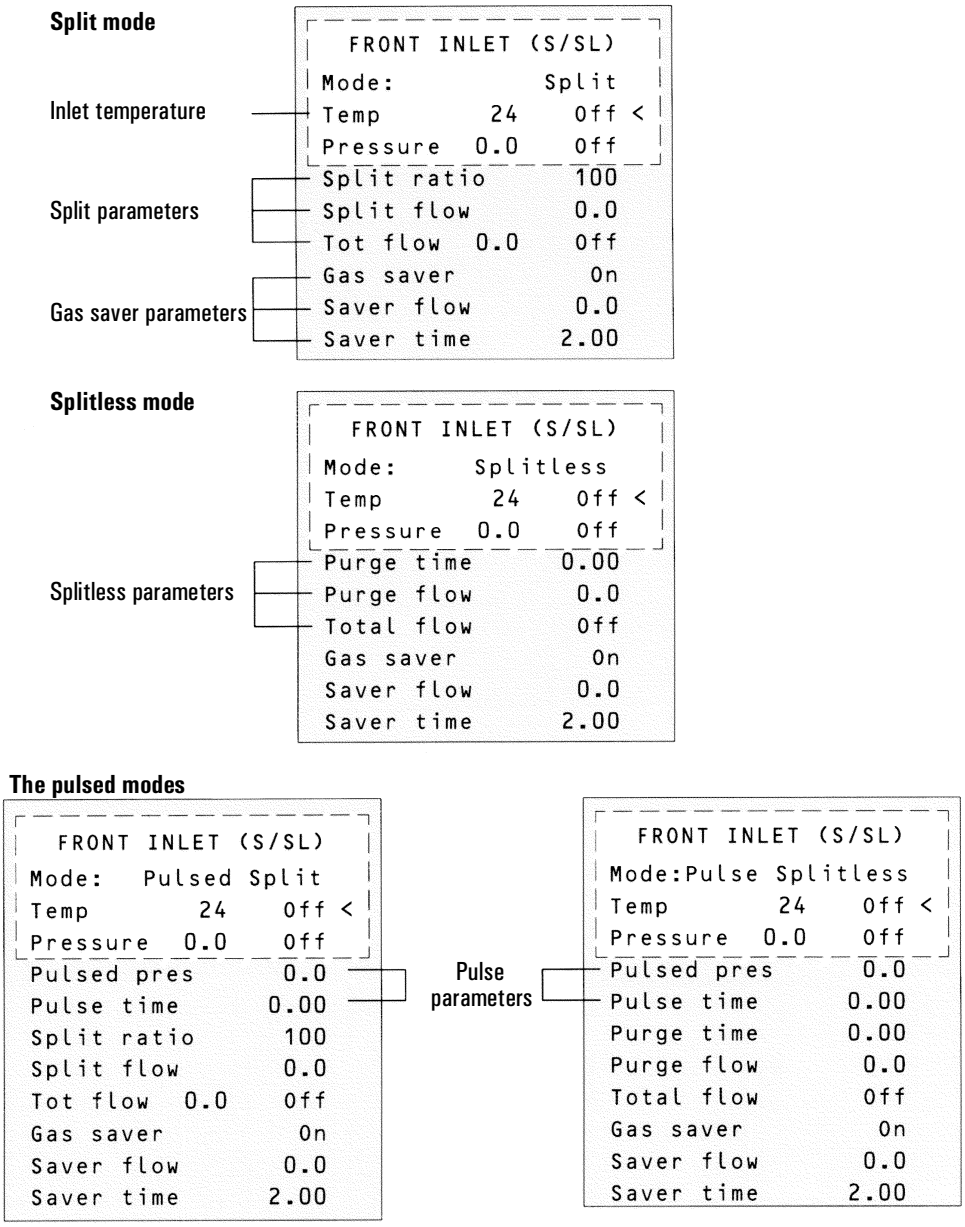


Figure 6 Control tables for the Split/Splitless inlet

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.

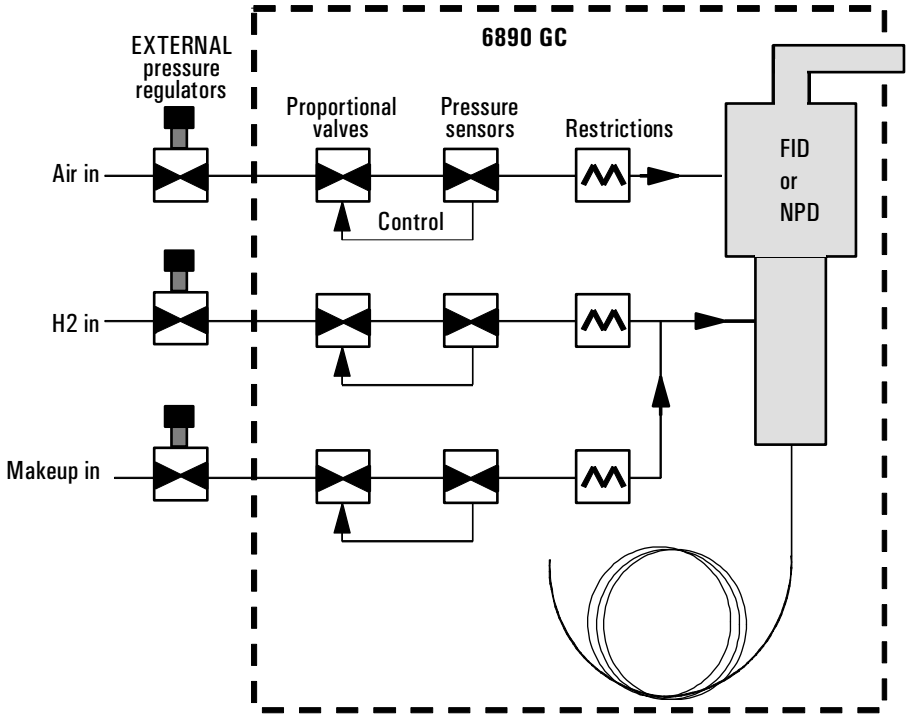


Figure 7 Internal/external plumbing: FID and NPD *with* EPC
For more detail, see "The Flame Ionization Detector", "The Nitrogen-Phosphorus Detector".

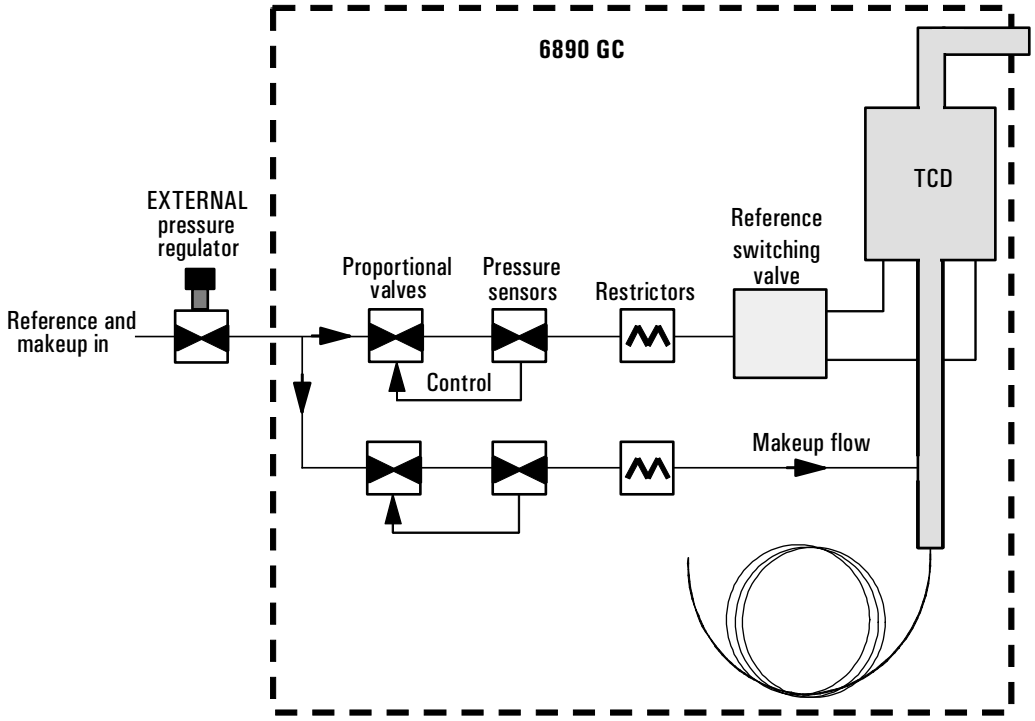


Figure 8 Internal/external plumbing: TCD *with* EPC
For more detail, see "The Thermal Conductivity Detector".

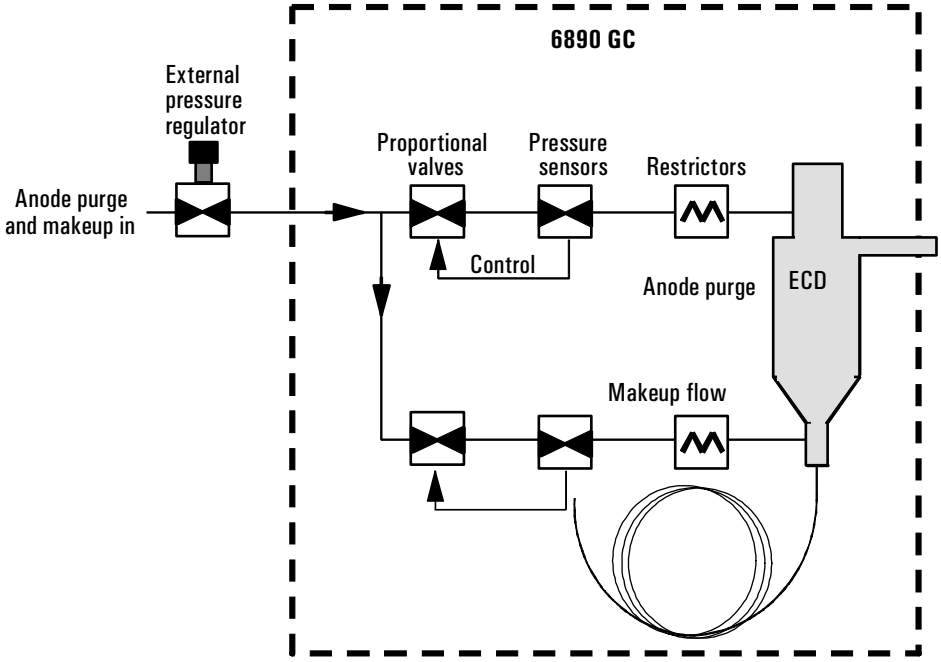


Figure 9 Internal/external plumbing: ECD *with* EPC
For more detail, see "The Micro-Cell Electron Capture Detector".

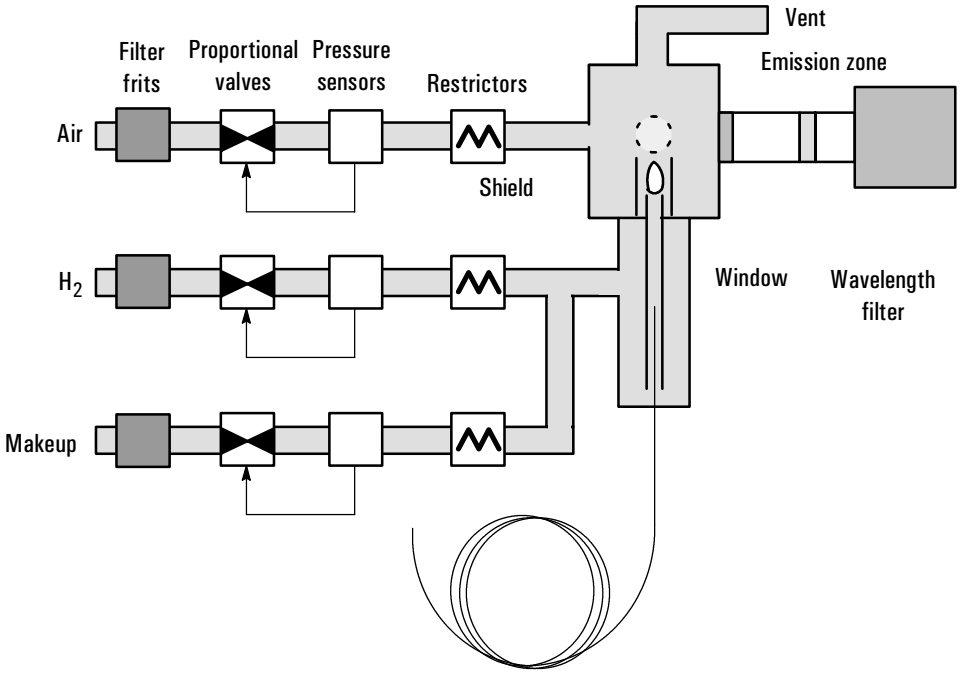


Figure 10 Internal/external plumbing: FPD *with* EPC
For more details, see "The Flame Photometric Detector".

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 Flame Ionization Detector", "The Thermal Conductivity Detector", "The Nitrogen-Phosphorus Detector", "The Micro-Cell Electron Capture Detector", "The Flame Photometric Detector" 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

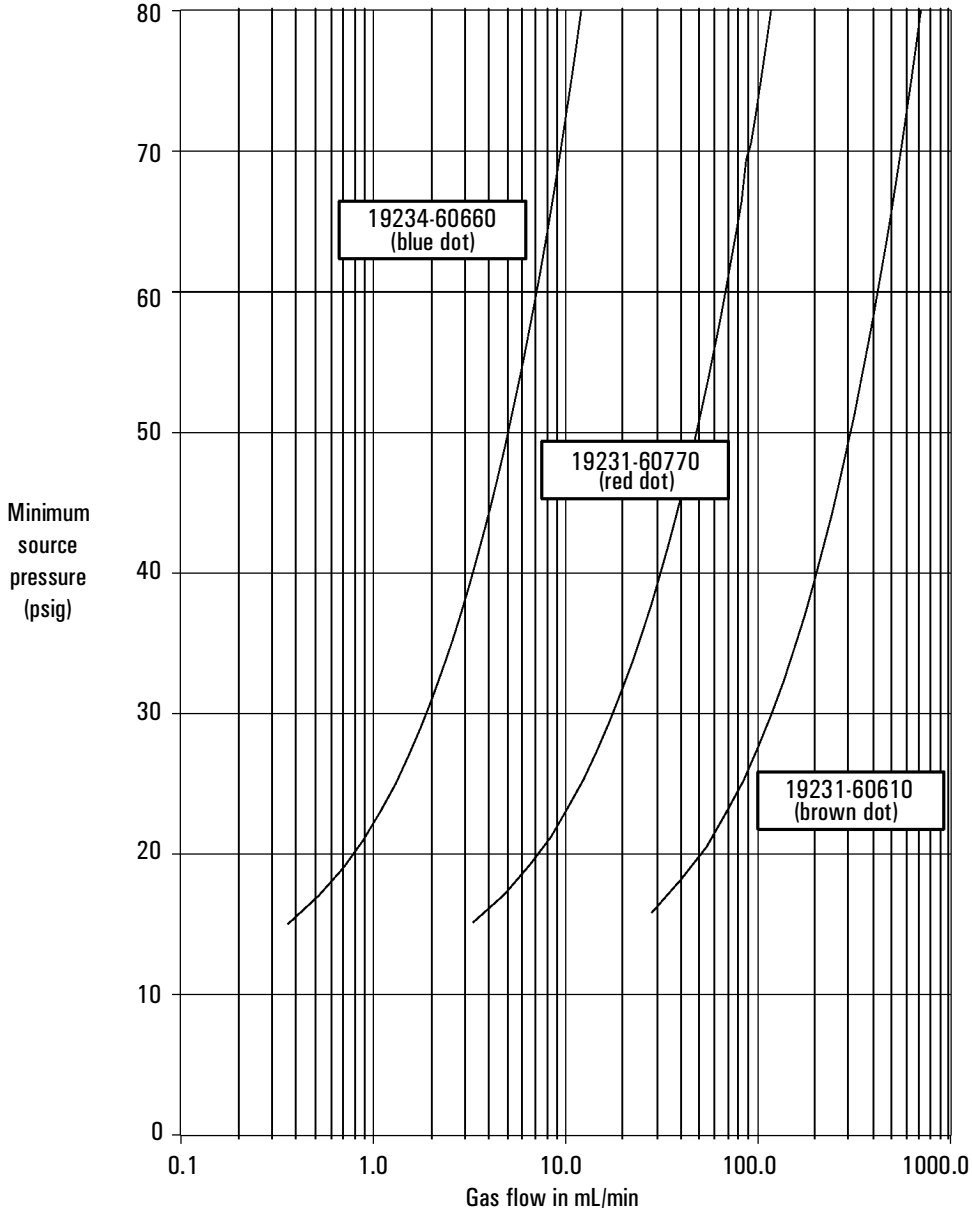
The Red Dot frit is in all three channels when the instrument is shipped.

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

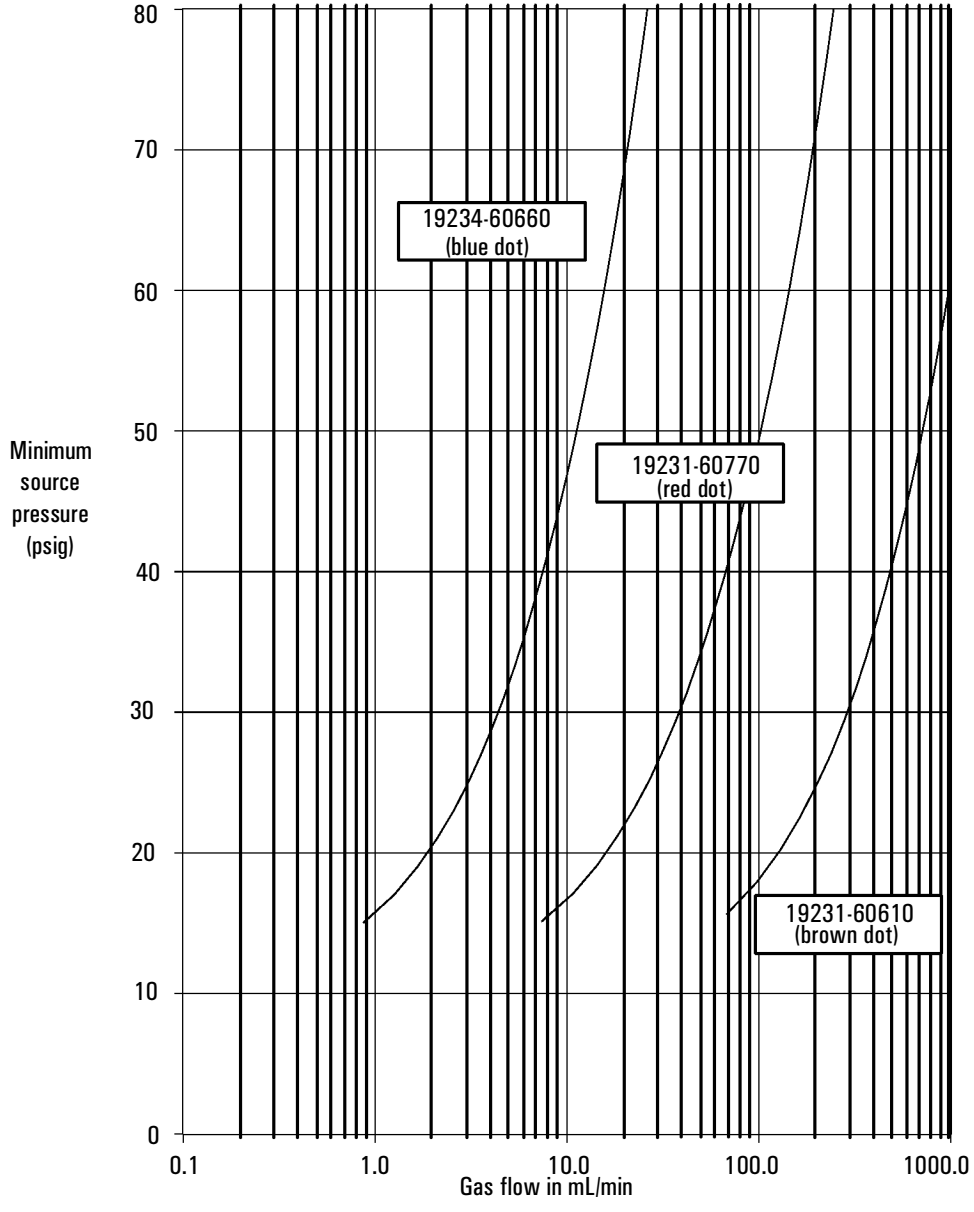
If the Zero resistance frit is installed, the user must provide flow resistance downstream and generate the pressure/flow relationships.

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)

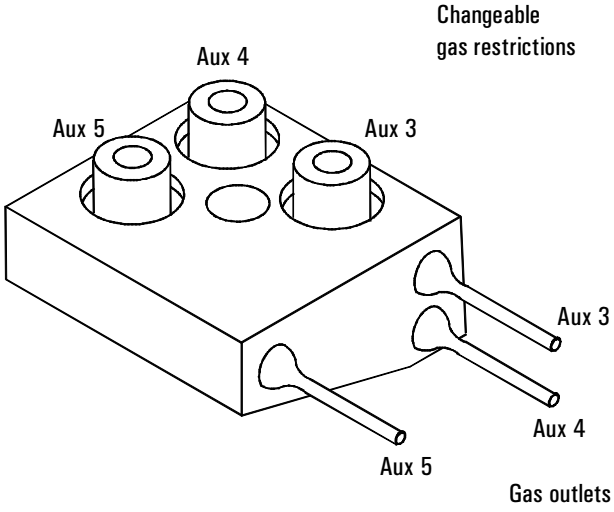


Pressure requirements for AUX EPC flow restrictors
with hydogen
(ambient conditions: 25°C, 14.7 psia)



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 54) 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.

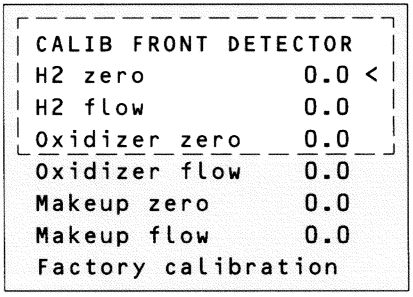
Table 9 Flow and Pressure Sensor Zero Intervals

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

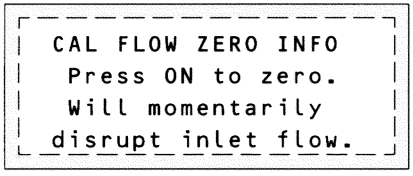
To zero a flow or pressure sensor in a specific module

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

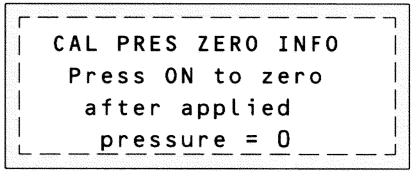


Note: After zeroing or flow calibration, the `Factory Calibration` line is replaced by the time and date of the recalibration.
To restore the Factory Calibration, select the time and date line and press [Delete]. This destroys the user calibration.

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



or

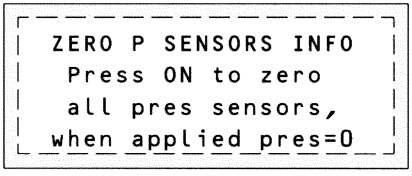


4. To cancel, press [Clear]
5. To zero flow, verify that the carrier gas is connected and is turned on.
6. Press [On] to zero or [Clear] to cancel.

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]



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.

NonEPC control

Control tables for nonEPC inlet 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 95 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 "Introduction to Inlets" for operating information.

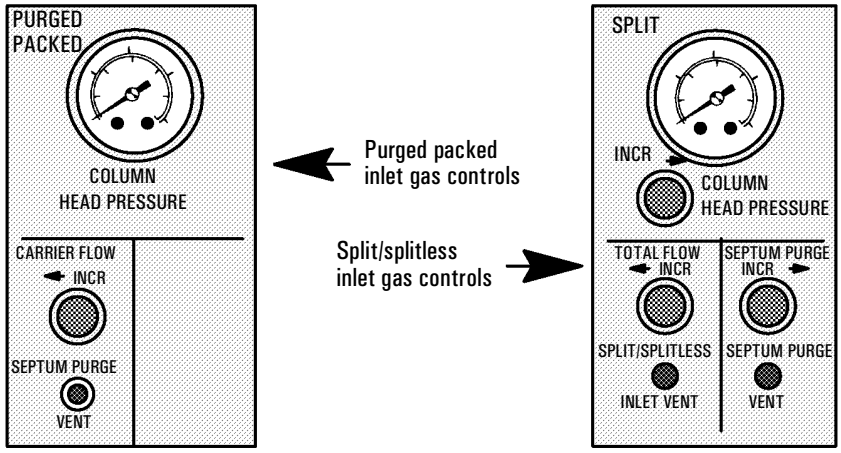


Figure 11 NonEPC inlet gas controls

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.

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 98) 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).

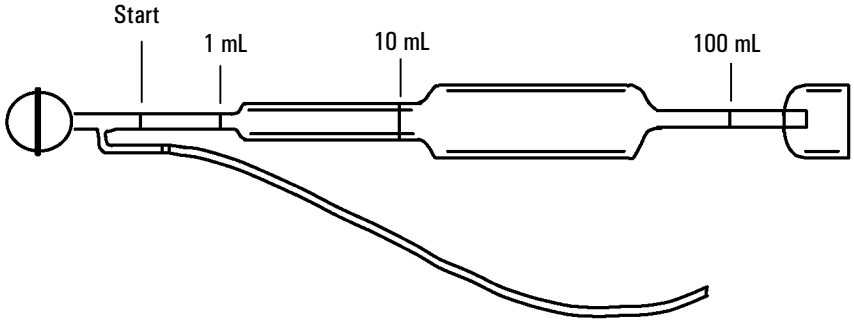
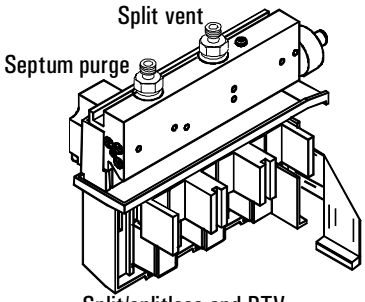


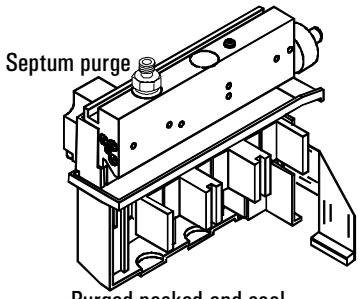
Figure 12 A three-volume bubble meter

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

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 Figure 11.

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 × 1/t.
   - If you used the 100 mL line, the flow rate in mL/min = 100 × 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).

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

Oven capabilities

Oven safety

Configuring the oven

Procedure: Setting up an isothermal run

Making a temperature-programmed run

Oven temperature programming setpoints

Oven ramp rates

Procedure: Setting up a single-ramp program

Procedure: Setting up a multiple-ramp program

Fast chromatography

Fast-heating oven

Configuring the oven

Using the oven insert for fast chromatography

To install the oven insert

Removing the insert

Cryogenic operation

Cryogenic control setpoints

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.

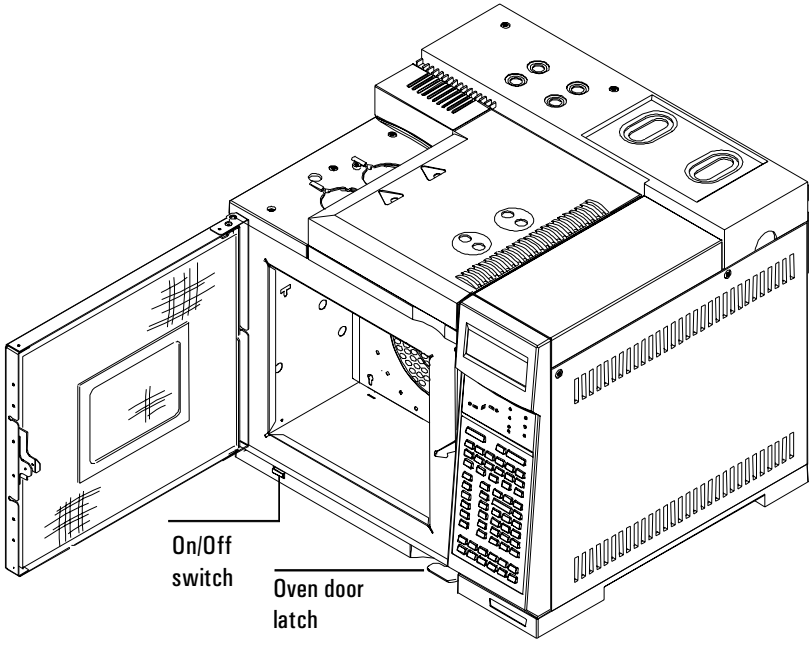


Figure 13 Column oven

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.

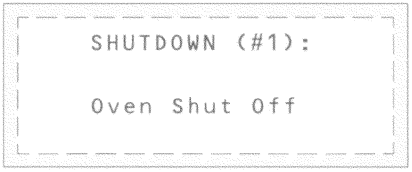
If the oven cannot attain or maintain an entered setpoint temperature during normal above-ambient operation, a problem is assumed and the oven is switched off.

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:



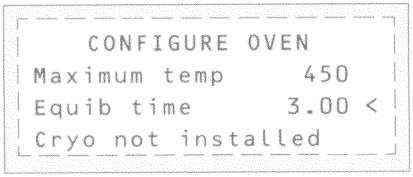
See "Shutdown" for more information on shutdowns.

Configuring the oven

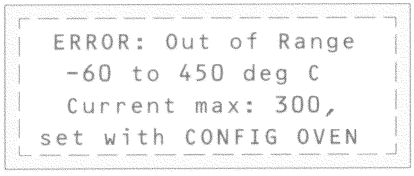
Oven configuration sets maximum temperature, equilibration time, and the cryogenic setpoints, if cryo is installed.

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.

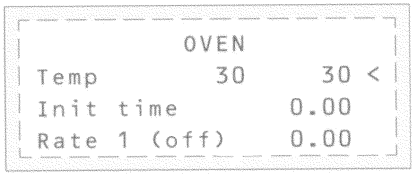


`Equib time` The time required for the oven temperature to equilibrate after temperature is modified. Equilibration time begins when the actual oven temperature comes within 1°C of the oven temperature setting. The `Equib time` setpoint can be 0 to 999.99 minutes.

Procedure: Setting up an isothermal run

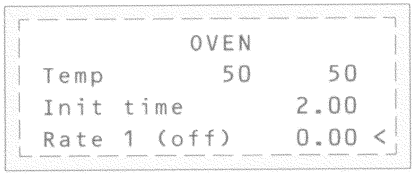
An isothermal run is one in which the oven is maintained at a constant temperature. To create an isothermal run, set `Rate 1` to zero.

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.

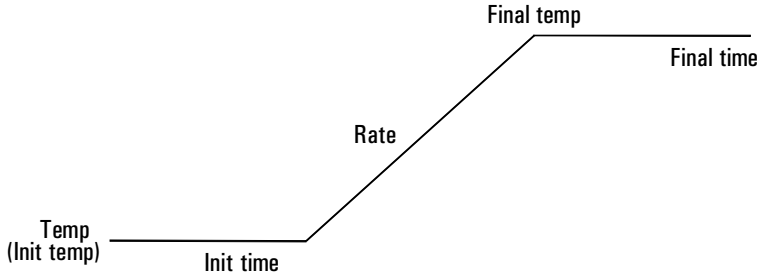


Figure 14 Single ramp

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.

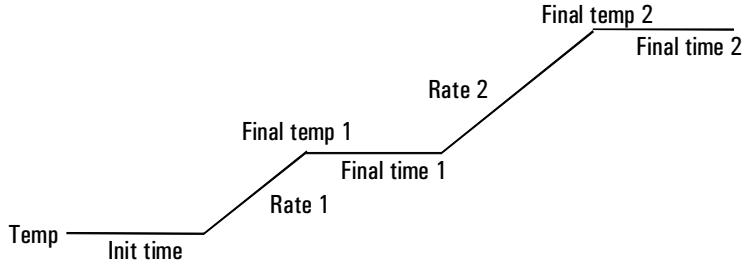


Figure 15 Multiple ramp

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 113), increases oven ramp rates for the back column. Table 10 lists typical oven ramp rates.

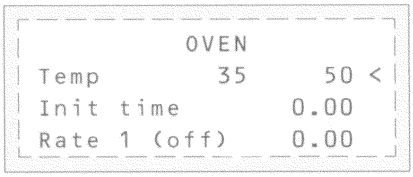
Table 10 Oven Ramp Rates

	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

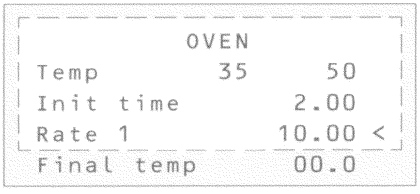
This example increases the oven temperature from 50°C to 150°C at a rate of 10°C/minute.

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 113), and cryogenic cooling (see page 116).

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.

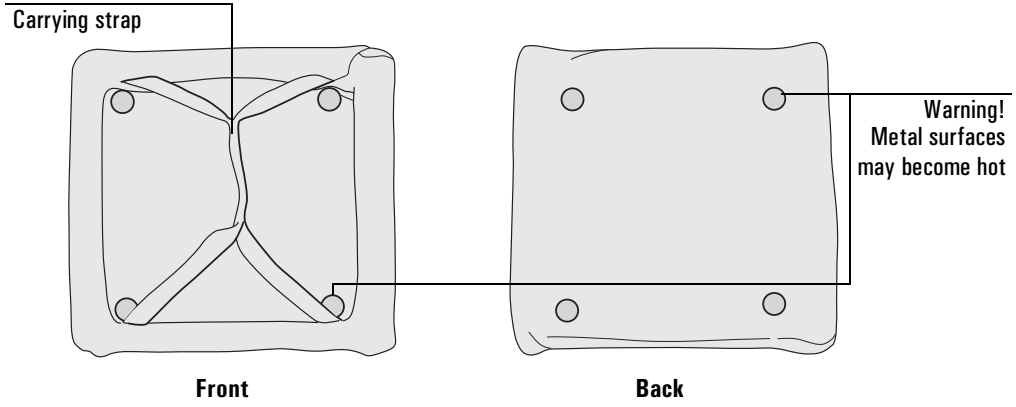


Figure 16 Oven insert

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 17 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.

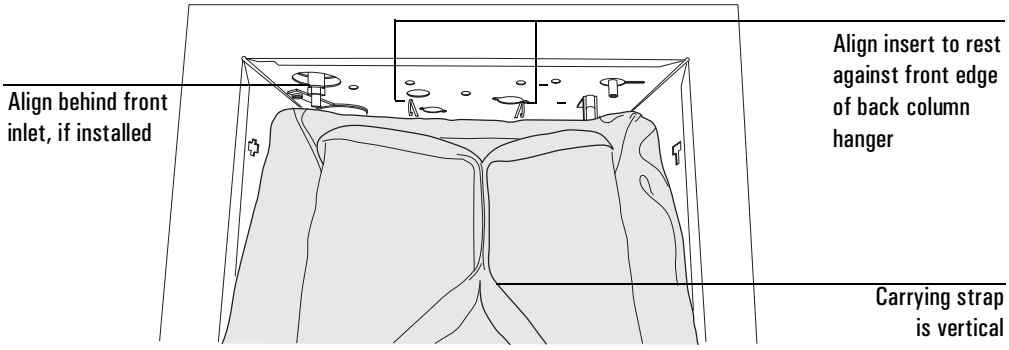


Figure 17 Installing the insert in front of the back column hanger

3. Push the bottom of the insert into place as shown in Figure 18. Keep the insert upright as shown.

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

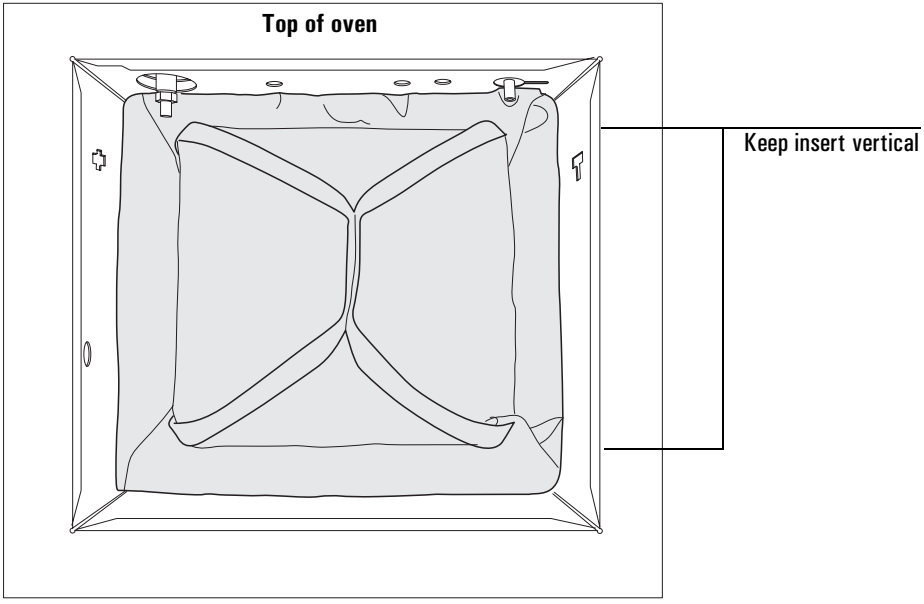


Figure 18 Oven insert installed in the oven

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

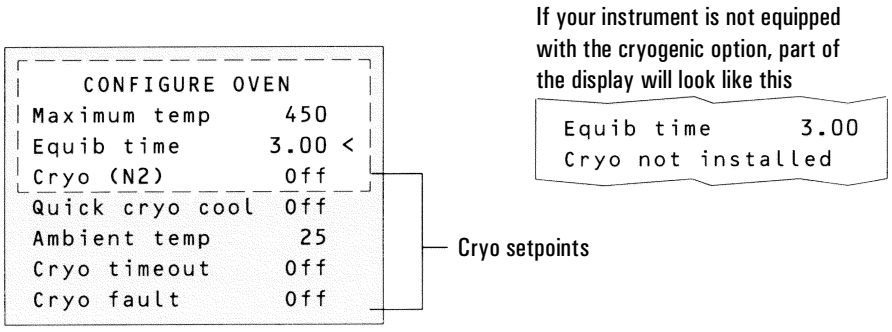
The cryogenic valve lets you operate the oven below ambient temperature. Minimum attainable oven temperature depends on the type of valve installed.

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".

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.

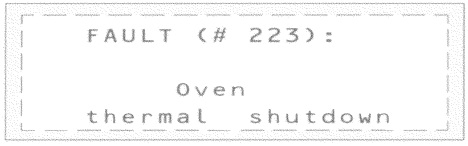
`Ambient temp` The temperature in the laboratory. This setpoint determines the temperature at which cryogenic cooling is enabled:

- 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

Capillary columns

Column hanger

Procedure: Preparing capillary columns

Procedure: Installing capillary columns in the split/splitless inlet

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

Procedure: Installing capillary columns in the purged packed inlet

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

Procedure: Installing capillary columns in NPD and FID detectors

Procedure: Installing capillary columns in the TCD

Procedure: Installing capillary columns in the μ-ECD

Procedure: Installing capillary columns in the FPD

Ferrules for capillary columns

Graphite and graphitized-Vespel ferrules

Vespel ferrules

Packed metal columns

Overview: installing packed metal columns

Fittings

Preparing packed metal columns

Procedure: Making a spacer from Teflon tubing

Procedure: Installing ferrules on a metal column

Procedure: Installing an adapter in a detector fitting

Procedure: Installing packed metal columns

Ferrules for packed metal columns

Packed glass columns

Overview: Installing glass packed columns

Procedure: Installing glass packed columns

Ferrules and O-rings for glass packed columns

Conditioning columns

Procedure: Preliminary column conditioning steps

Procedure: Conditioning a capillary column
Procedure: Conditioning packed columns

Conditioning chemical traps

Calibrating your capillary column (optional)

Calibration modes

Column calibration procedures

Procedure: Estimate the actual column length or diameter from an elution time
Procedure: Estimate the actual column length or diameter from the measured flow rate
Procedure: Estimate the actual column length and diameter

Columns and Traps

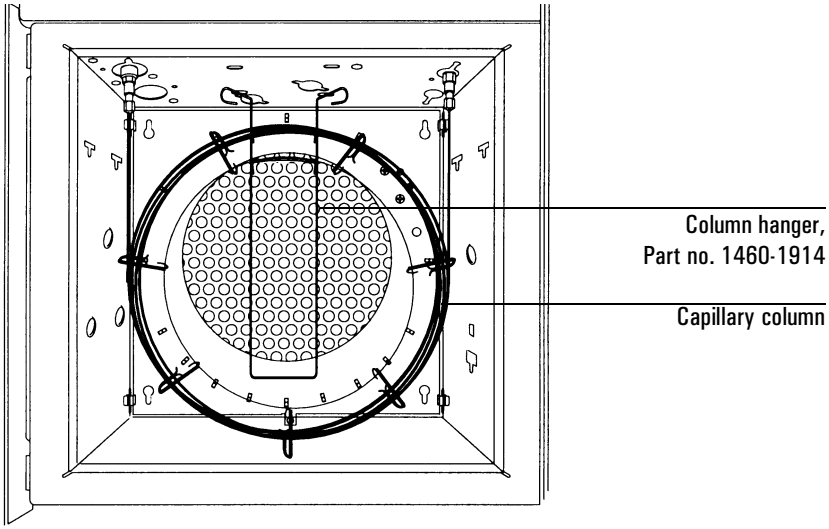
Capillary columns

This section contains information on preparing and installing capillary columns in inlets and detectors. See page 141 for packed metal columns and page 150 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

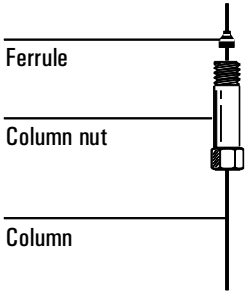
You must prepare your capillary column before installation. Proper preparation assures that the column end has no burrs or jagged edges and is not contaminated with graphite or other material.

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.

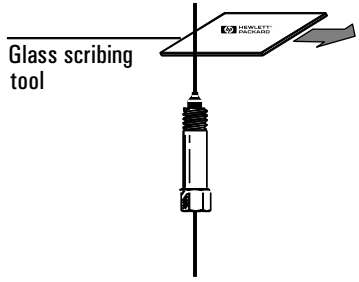
Materials required

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

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

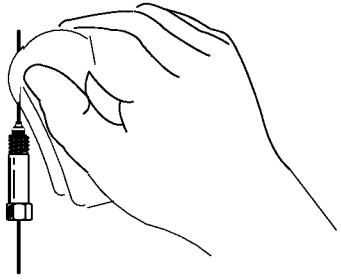


2. Score the column using a glass scribing tool. The score must be square to ensure a clean break.



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.



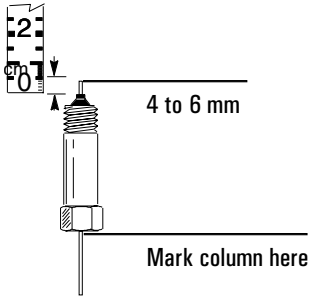
Procedure: Installing capillary columns in the split/splitless inlet

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

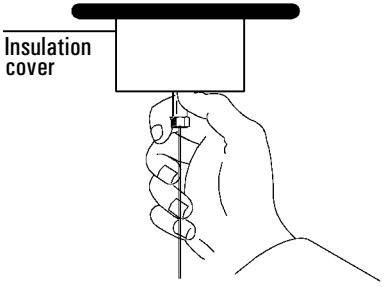
Materials required

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

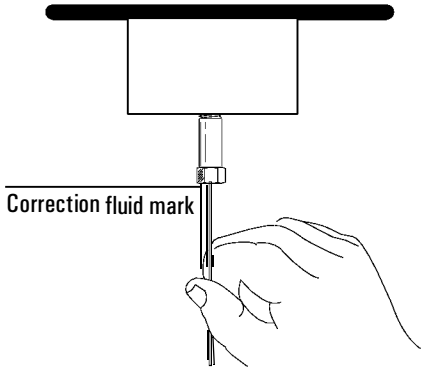
1. Prepare the column. See page 121 for instructions.
2. Position the column so it extends 4 to 6 mm above the end of the ferrule. Mark the column with typewriter correction fluid at a point even with the column nut.



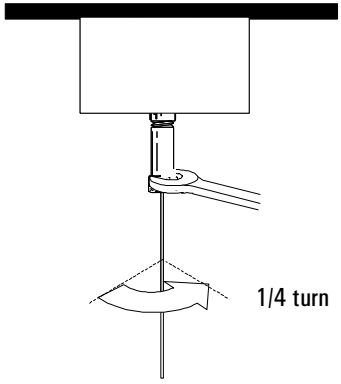
3. Insert the column in the inlet and slide the nut and ferrule up the column to the inlet base. Finger tighten the column nut until it starts to grab the column.



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.

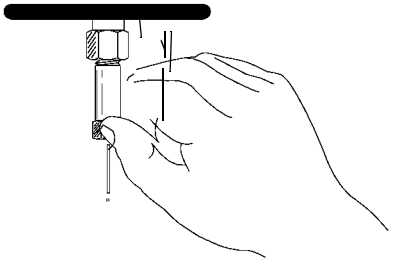
Procedure: Installing capillary columns in the cool on-column inlet

Before installing the column, be certain you have the correct hardware installed for the column and type of injection you are doing. See "Hardware for the cool on-column inlet" for detailed information.

Materials required

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

1. Prepare the column. See page 121 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.

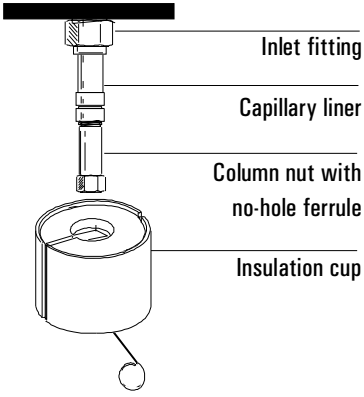
Procedure: Installing capillary columns in the purged packed inlet

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 "Liners and inserts". If your insulation cup is not installed, begin with Step 1. Otherwise, begin with Step 4.

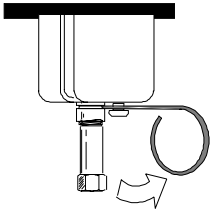
Materials required

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

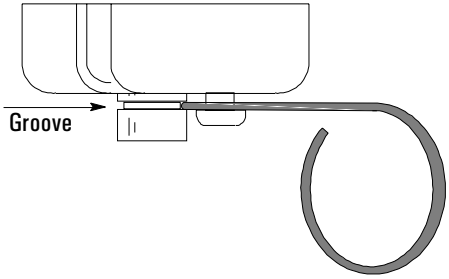
1. Install a plug in the inlet fitting.



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.

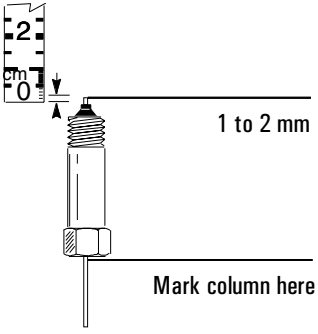


3. Place the spring into the groove in the inlet liner. Remove the column nut and put the no-hole ferrule aside.

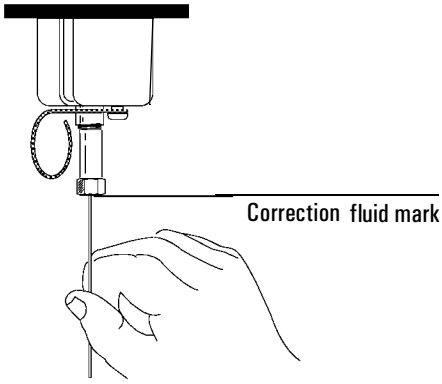


4. Prepare the column. See page 121 for instructions.

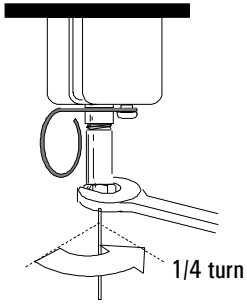
5. Position the column so it extends above the end of the column nut by 1 to 2 mm. Mark the column with typewriter correction fluid at a point even with the column nut.



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 "Procedure: Installing columns" and "Procedure: Installing columns".

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

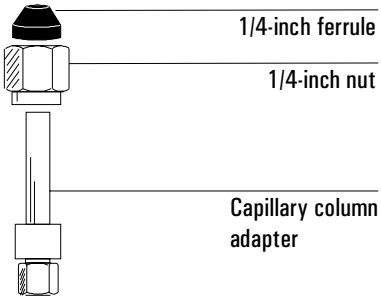
Both fitting types:

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

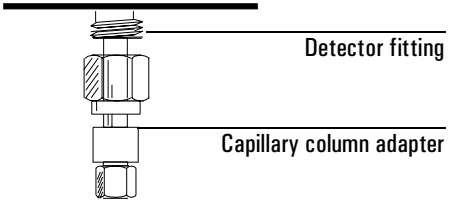
For adaptable fitting only:

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.



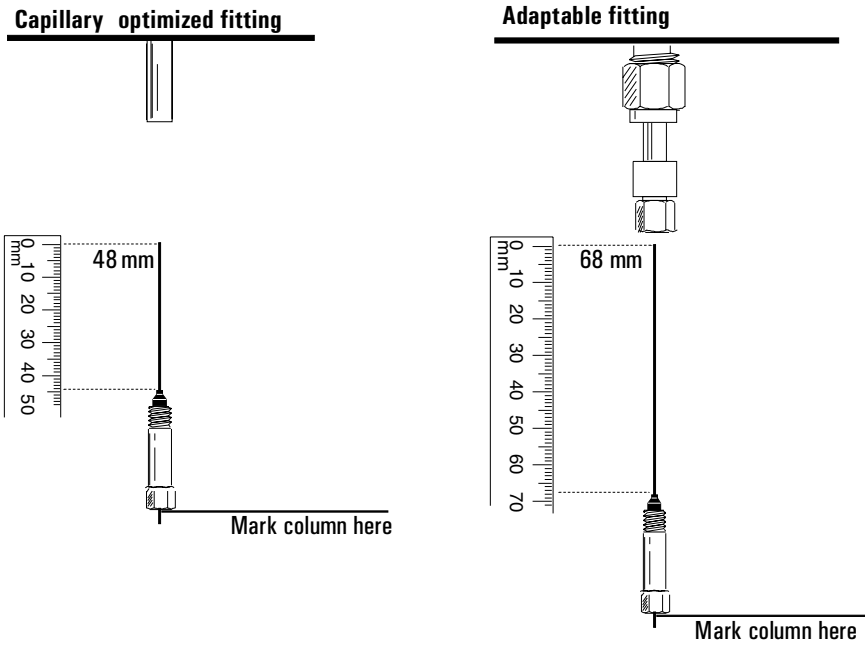
2. Insert the adapter straight into the detector base as far as possible. Hold the adapter in this position and tighten the nut finger tight. Use a wrench to tighten the nut an additional 1/4 turn.



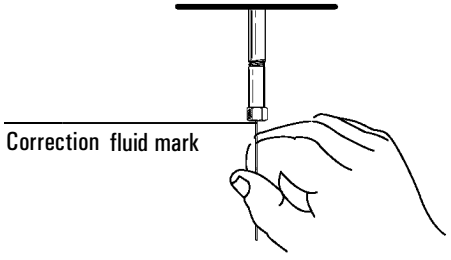
3. Prepare the column (see page 121 for instructions). If the column id is greater than 100 µm, follow steps 7 to 9. If the column id is less than 100 µm, follow steps 4 to 6, then follow steps 8 and 9.

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.

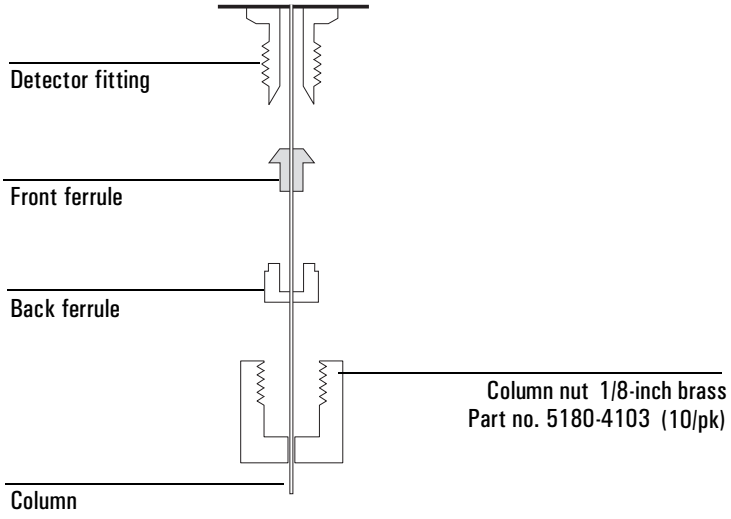
8. Tighten the column nut finger tight, then withdraw the column about 1 mm. Use a wrench to tighten the nut an additional 1/4 turn.
9. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures.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

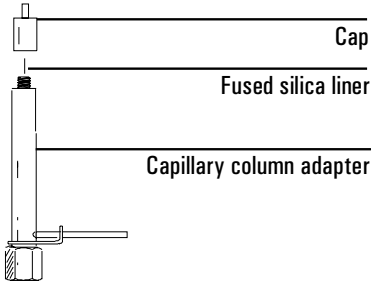
The detector is shipped with a capillary column adapter installed. If it has been removed, you must replace it before installing a capillary column.

The μ-ECD requires the indented liner, which is necked down near one end and is clear.

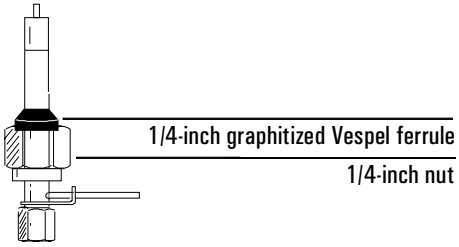
Materials required

Capillary column adapter
Fused silica liner, indented
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. The indentation must be at the cap end of the adapter.



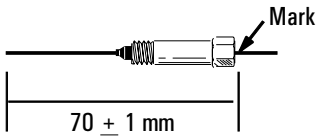
2. Install a 1/4-inch nut and graphitized-Vespel ferrule on the adapter.



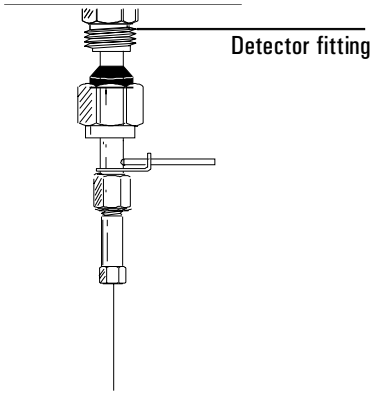
3. Prepare the column. See page 121 for instructions.

4. 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.

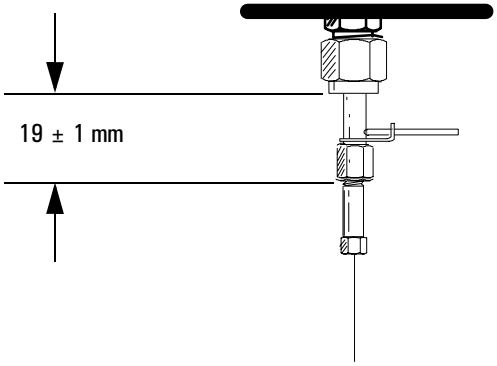
   If the id is less than 200 µm, mark the column 70±1 mm from the end. Insert column and nut into the adapter with the mark at the rear of the column nut, and tighten the column nut firmly.



5. 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.

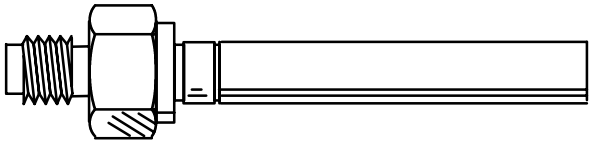


6. Slide the nut and ferrule up to the detector fitting and tighten the nut finger tight. Use a 9/16-inch wrench to tighten the nut an additional 1/4 turn.
7. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector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

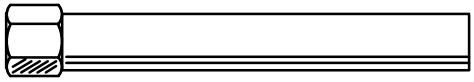
Procedure: Installing capillary columns in the FPD

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 µm 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

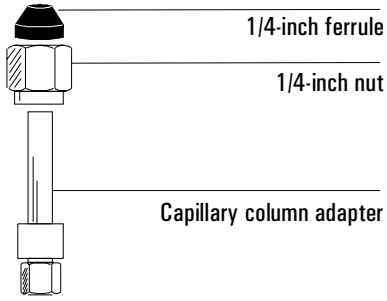


19256-80570
FPD Capillary Adapter

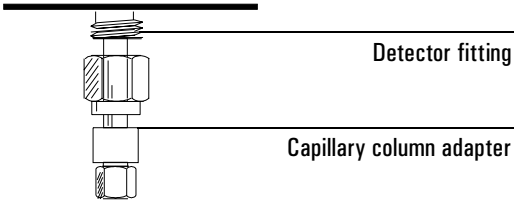
Materials required

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.



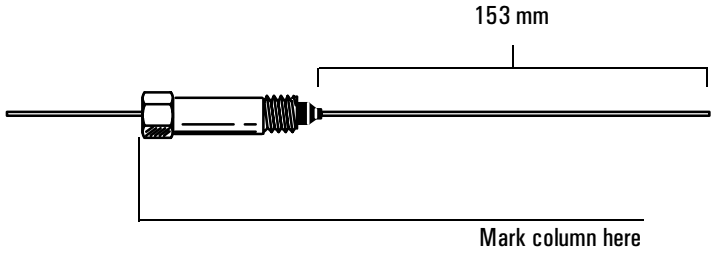
2. Insert the adapter straight into the detector base as far as possible. Hold the adapter in this position and tighten the nut finger tight. Use a wrench to tighten the nut an additional 1/4 turn.



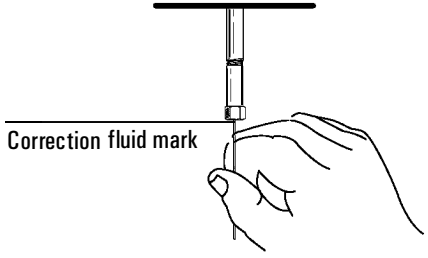
3. Install a column nut (part no. 18740-20870) and graphite ferrule (1.0 mm ID, part no. 5080-8773 or 0.5 mm ID, part no. 5080-8853) on the column.
4. After installing the nut and ferrule, prepare a fresh column end by cutting off a short piece of the column. See page 121 for instructions.
5. Position 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

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 143).
2. See Table 13 or Table 14 for fittings required.
3. Install the adapter, if needed (page 147).
4. Install the column (page 148).
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 *"Procedure: Installing liners"* for instructions on installing liner.	1/8-inch liner	See *"Procedure: Installing liners"* 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 147 for instructions on installing an adapter.	1/8-inch adapter (Part no. 19231-80520)	See page 147 for instructions on installing an adapter.
ECD	Detector fitting	Remove adapter, if necessary.	1/8-inch adapter. (Part no. 19301-80530)	See page 147 for instructions on installing an adapter.
TCD	1/4-inch adapter (Part no. G1532-20710)	See page 147 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.

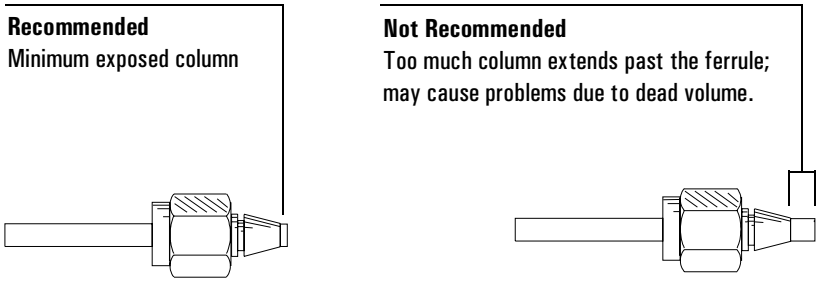
The FPD 1/8-inch OD Adapter, part number 19256-80590, allows installation of PTFE columns concentrically around the FPD fused silica liner, eliminating exposed hot stainless steel.

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 147 for instructions on installing an adapter.	1/8-inch adapter (Part no. 19256-80590)	See page 147 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.



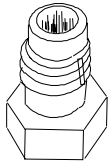
Use the following instructions to install new SWAGELOK nuts and ferrules onto 1/8-inch or 1/4-inch metal columns. If your column already has ferrules installed, proceed to the instructions on installing adapters (page 147) or installing packed metal columns (page 148).

Procedure: Making a spacer from Teflon tubing

Materials required

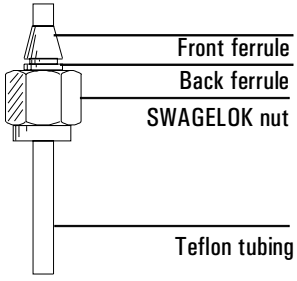
1/4-inch or 1/8-inch Teflon tubing
1/4-inch or 1/8-inch nut and ferrule set
Bench vise
Male SWAGELOK fitting
9/16-inch or 7/16-inch wrench
Razor or sharp knife

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

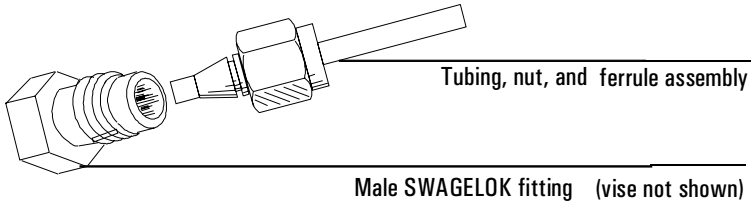


Male SWAGELOK fitting

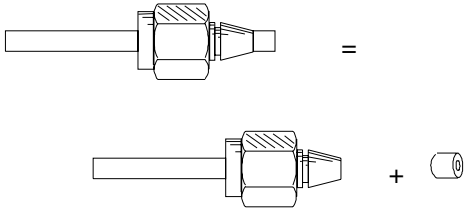
2. Slide a SWAGELOK nut, back ferrule, and front ferrule onto a piece of Teflon tubing. If the end of the tubing is not cut straight, use a razor or sharp knife to make a flat, smooth end.



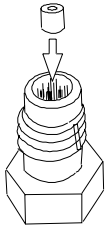
3. Insert the Teflon tubing, ferrules, and nut into the vise-held SWAGELOK fitting. Tighten the nut 3/4 turn past finger tight to set the ferrules on the tubing.



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.



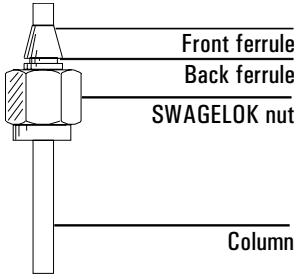
The male SWAGELOK fitting and spacer should be kept on hand to be used whenever new ferrules are being installed on a column.

Procedure: Installing ferrules on a metal column

Materials required

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

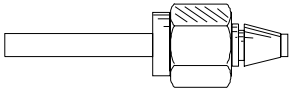
1. Install new SWAGELOK nut and ferrules on the column.



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.



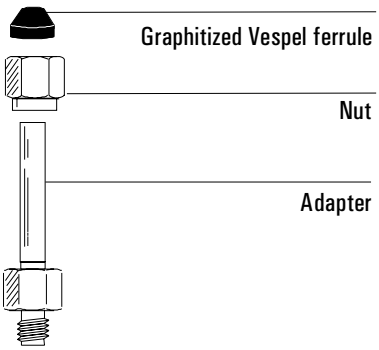
Procedure: Installing an adapter in a detector fitting

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

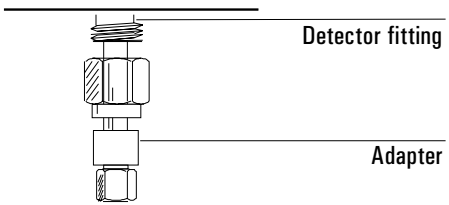
Materials required

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 148.

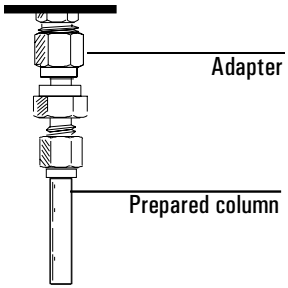
Procedure: Installing packed metal columns

Before following this procedure, make sure an adapter or liner is installed (page 147), if needed, and that your column is prepared (page 143.)

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.

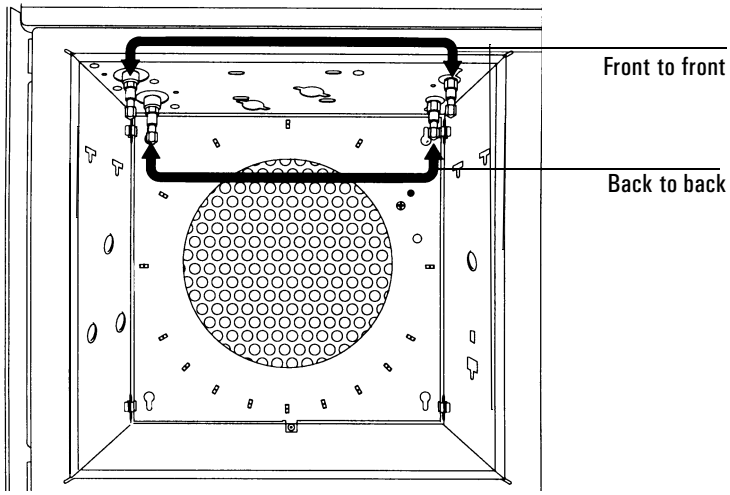
Table 15 Nuts and Ferrules Used with Packed Metal Columns

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

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, µ-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 147).
3. Follow the general procedure for installing glass columns on page 152.

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.
μ-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 147.

* See the "Procedure: Installing liners" for instructions on installing liners

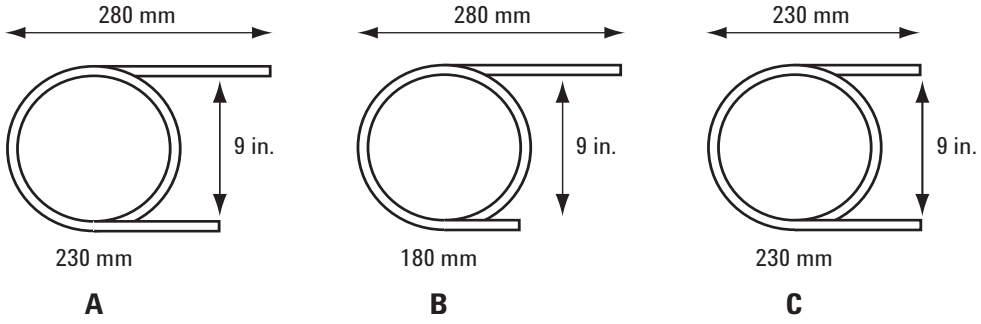


Figure 19. Column Configurations

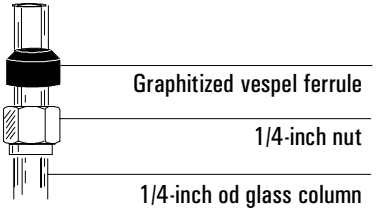
Procedure: Installing glass packed columns

Materials required

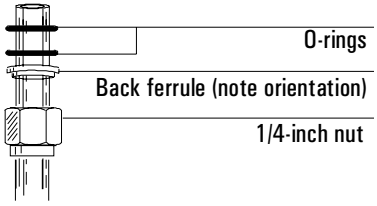
Recommended:	**Alternative:**
Two 1/4-inch graphitized Vespel ferrules	Four O-rings
Two 1/4-inch nuts	Two back ferrules
9/16-inch wrench	Two 1/4-inch nuts
	9/16-inch wrench

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.

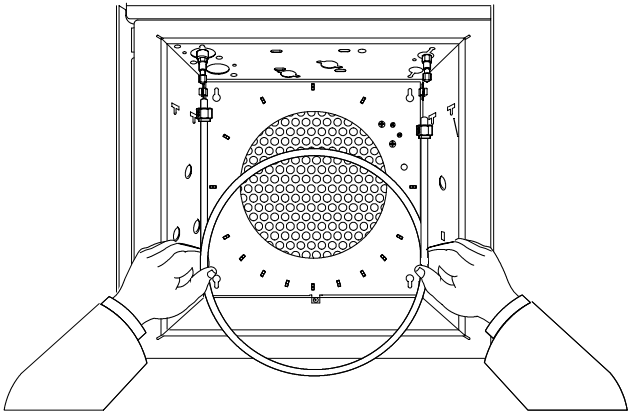
Recommended



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

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.

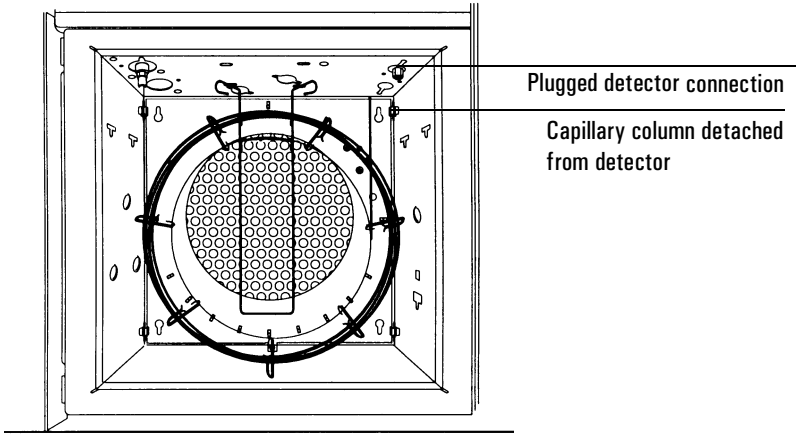
The following procedures include preliminary steps and the actual conditioning procedure, which differs for packed and capillary columns.

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.

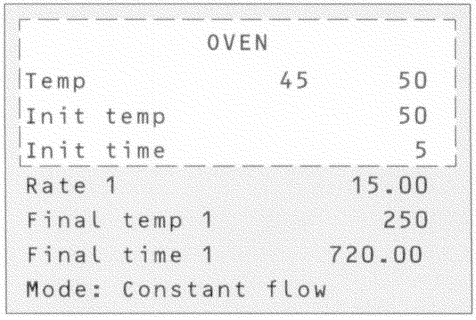
	Inside diameter				
Length, m	**0.10 mm**	**0.20 mm**	**0.25 mm**	**0.32 mm**	**0.53 mm**
10	25 (170)	6 (40)	3.7 (26)	2.3 (16)	0.9 (6.4)
15	39 (270)	9 (61)	5.6 (39)	3.4 (24)	1.4 (9.7)
25	68 (470)	15 (104)	9.5 (65)	5.7 (40)	2.3 (16)
30	83 (570)	18 (126)	12 (80)	7 (48)	2.8 (19)
50		32 (220)	20 (135)	12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

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

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

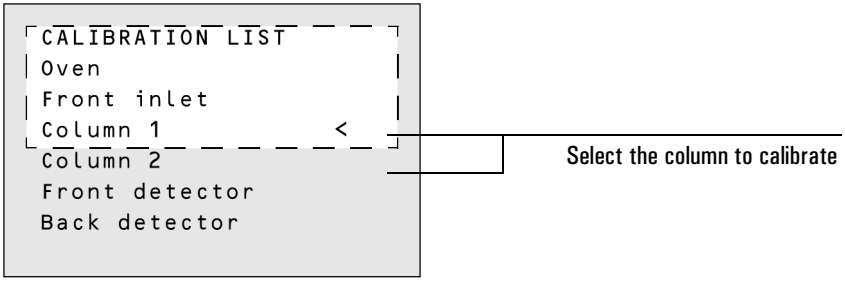
Caution When you measure the column flow rate, be sure to convert the measurement to normal temperature and pressure if your measurement device does not report data at NTP. If you enter uncorrected data, the calibration will be wrong. See "Measuring flow rates with a bubble meter" for details.

Column calibration procedures

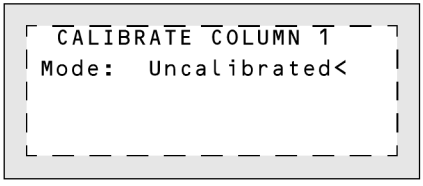
These procedures are described below using Column 1 as an example.

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

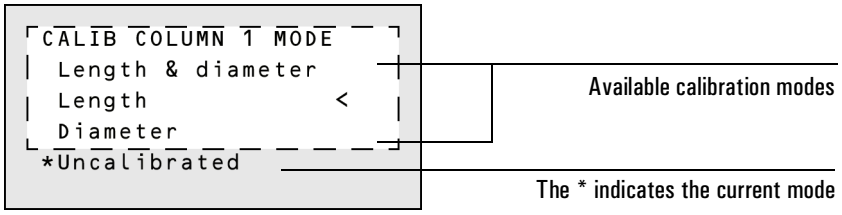
1. Set oven ramp 1 to `0.00`, then verify that the column is defined. For more information, see "Procedure: Setting up an isothermal run" or "Configure the column".
2. Perform a run using an unretained compound and record the elution time.
3. Press [Options]. Scroll to `Calibration` and press [Enter].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



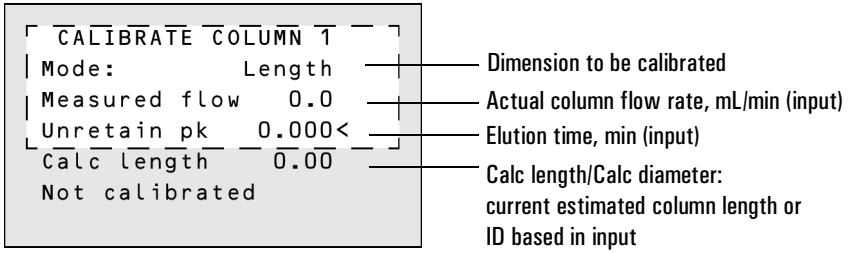
5. The GC displays the current calibration mode for the column. In this example, the column is uncalibrated.



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.

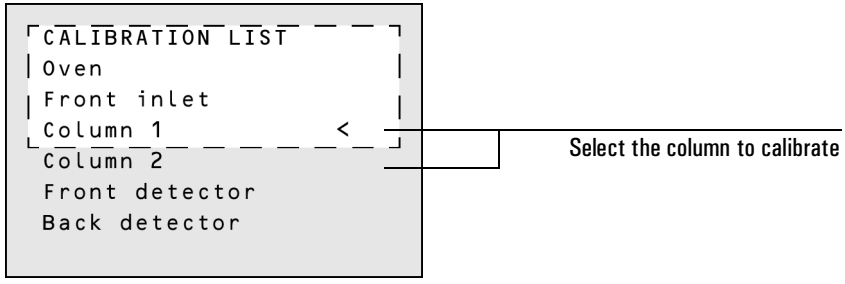
9. When you press [Enter], the GC will estimate the column length or diameter based on the elution time input and will now use that data for all calculations.

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

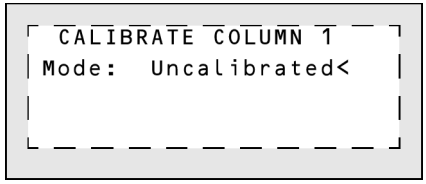
1. Set oven ramp 1 to `0.00`, then verify that the column is defined. For more information, see "Procedure: Setting up an isothermal run" or "Configure the column".
2. Set the oven, inlet, and detectors temperatures to 35°C and allow them to cool to room temperature.
3. Remove the column from the detector.

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 "Measuring flow rates with a bubble meter" for details.

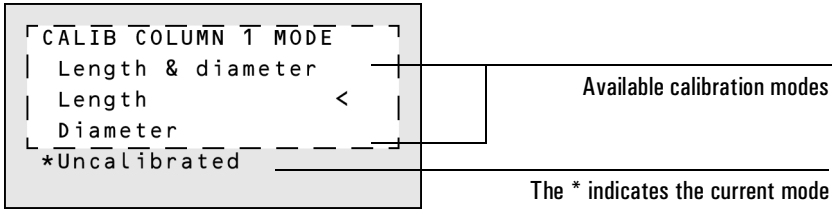
4. Measure the actual flow rate through the column using a bubble meter. Record the value. Reinstall the column.
5. Press [Options]. Scroll to `Calibration` and press [Enter].
6. From the calibration list, select `Column 1` or `Column 2` and press [Enter].



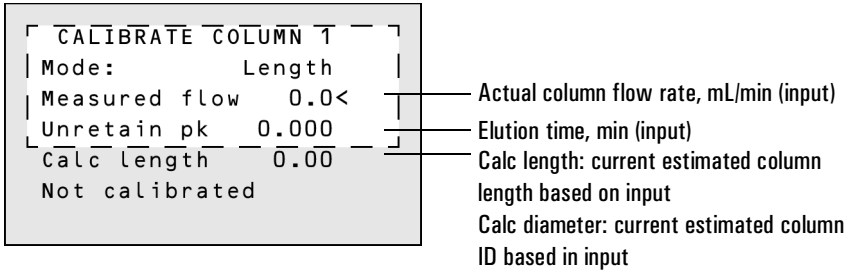
7. The GC displays the current calibration mode for the column. In this example, the column is uncalibrated.



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.

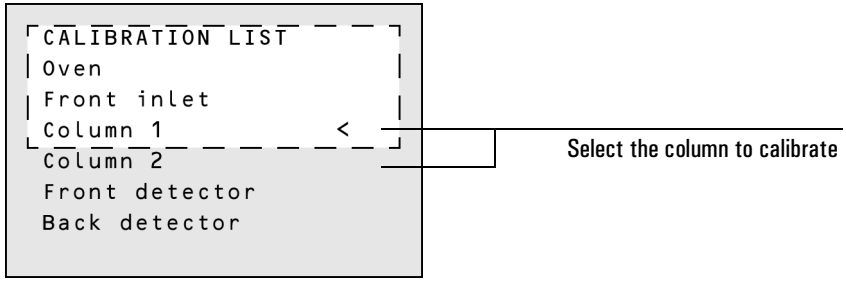
11. When you press [Enter], the GC will estimate the column length or diameter based on the elution time input and will now use that data for all calculations.

Procedure: Estimate the actual column length and diameter

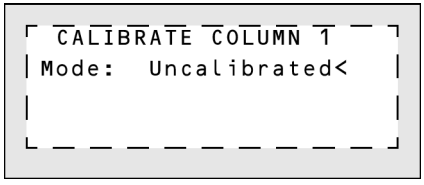
1. Set oven ramp 1 to `0.00`, then verify that the column is defined. For more information, see "Procedure: Setting up an isothermal run" or "Configure the column".
2. Perform a run using an unretained compound and record the elution time.
3. Set the oven, inlet, and detectors temperatures to 35°C and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

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 "Measuring flow rates with a bubble meter" for details.

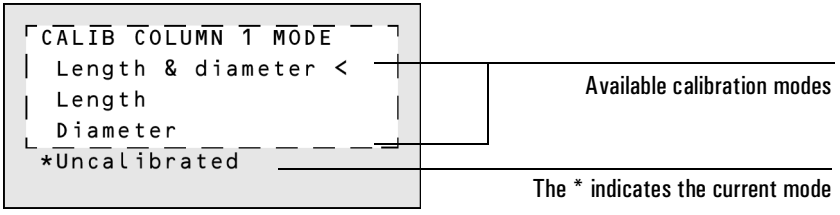
5. Measure the actual flow rate through the column using a bubble meter. Record the value. Reinstall the column.
6. Press [Options]. Scroll to `Calibration` and press [Enter].
7. From the calibration list, select `Column 1` or `Column 2` and press [Enter].



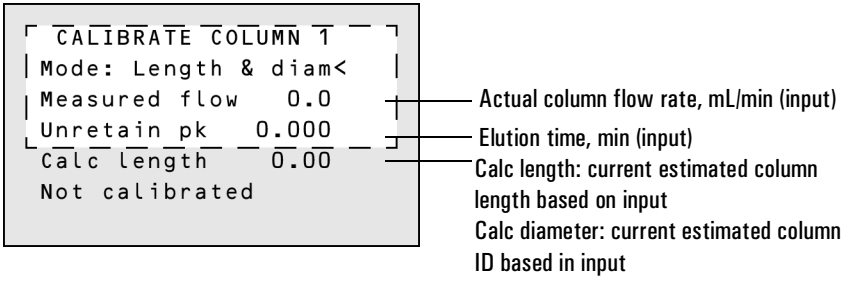
8. The GC displays the current calibration mode for the column. In this example, the column is uncalibrated.



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

Using the signal control tables
Signal type
Value

Analog output settings—zero, range, and attenuation
Analog zero
Procedure: Zeroing signal output
Range—for analog outputs only
Attenuation—for analog outputs only
Data rates
Procedure: Selecting fast peaks

Digital data handling
Digital zero
Baseline level shifts
Cerity/ChemStation

Column compensation
Procedure: Creating a column compensation profile
Procedure: Making a run using column compensation
Procedure: Plotting a stored column compensation profile

Test plot

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 Cerity and ChemStation software 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 Cerity or ChemStation software.

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 "Run time programming" for details.

Value

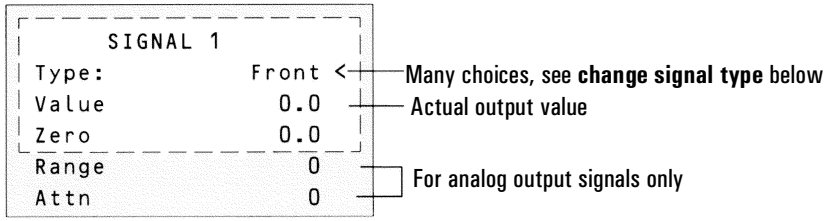
`Value` on the signal control table is the same as Output on the detector control table if your signal type is `Front` or `Back`. If you are subtracting one signal from another (as in `Front - Back`), the signal `Value` will be the difference. You cannot enter a setpoint for `Value`.

A conversion factor may be involved when interpreting `Value`—for example, one FID unit is one picoamp; one ECD unit is 1 Hz. 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)
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]:

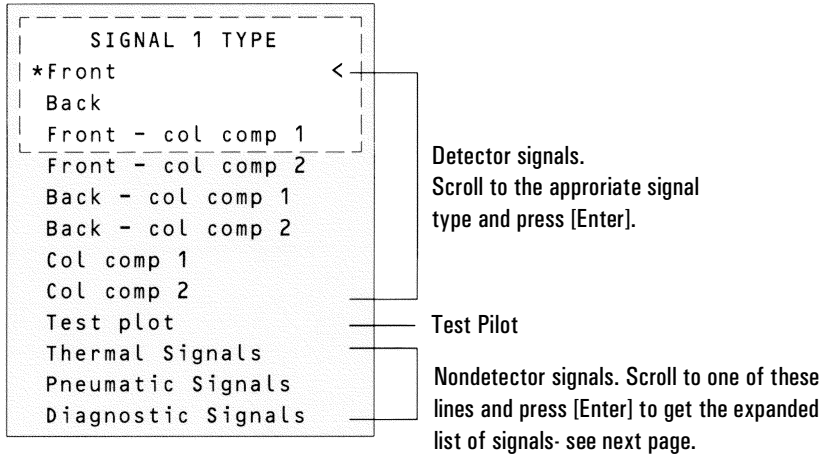
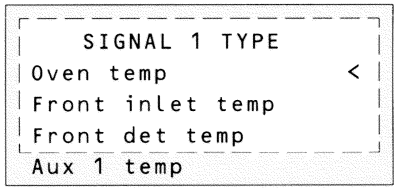
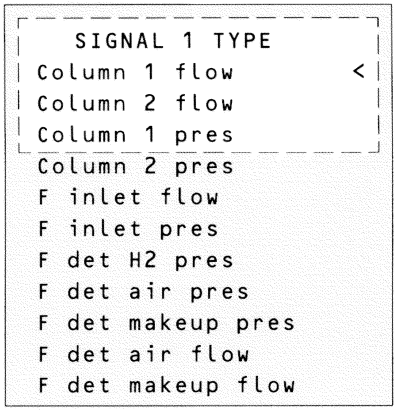


Figure 20 Signal control table

Thermal signals:

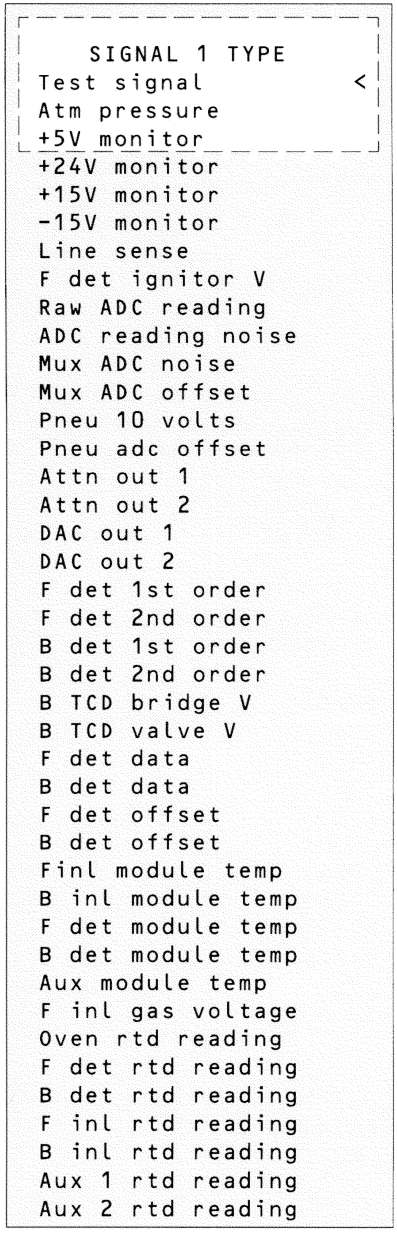


Pneumatic signals:



Only installed items are listed in submenus.

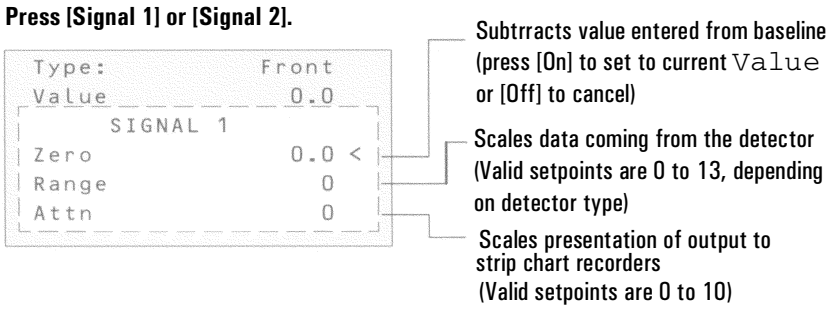
Diagnostic signals:



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].



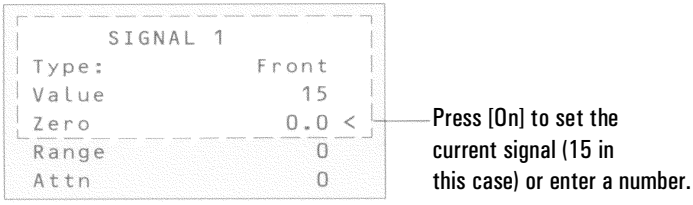
Analog zero

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 "Procedure: Programming run time events".

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.).

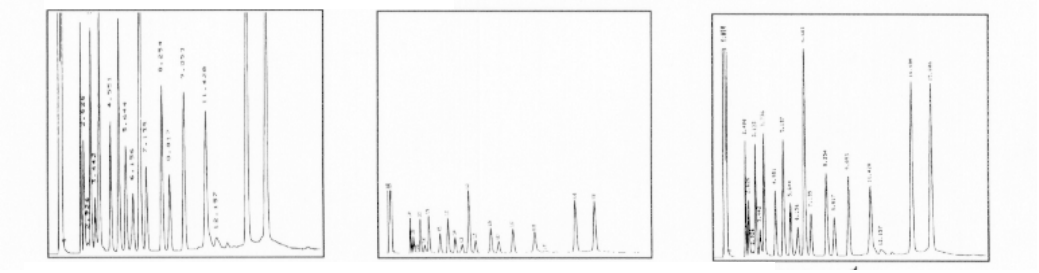
If a chromatogram looks like A or B in Figure 21, the data needs to be scaled (as in C) so that all peaks are visible on the paper.

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 21 illustrate this. Use the smallest possible value to minimize integration error.

See Table 20 for output scaling.

Table 20 Output Scaling

Analog	One display unit =	Digital	One display count =
0 to 1 mV	$1mV/(2^{Range} \times 2^{Attn})$	Cerity and ChemStation software	1 height count



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

Figure 21 Effect of range setting on chromatogram

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
μ-ECD	0 to 6
Analog input	0 to 7

Range may be run time programmed. See "Procedure: Programming run time events" 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 "Procedure: Programming run time events" 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 to minimize such problems—see "Run time programming".

`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 22.

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.

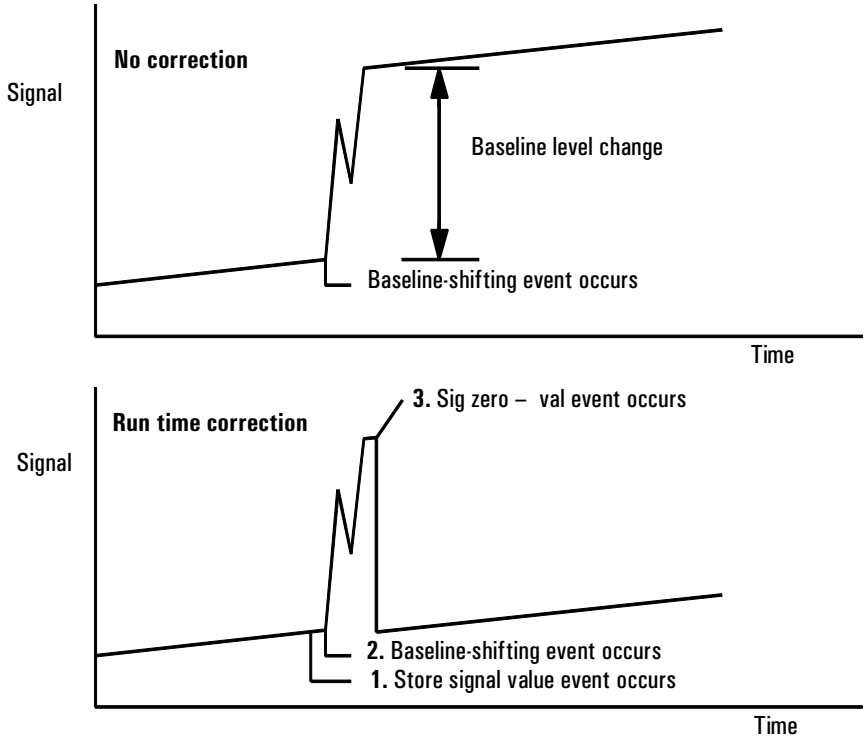


Figure 22 Correcting baseline level shifts in digital signals

Cerity\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 Cerity\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 23 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.

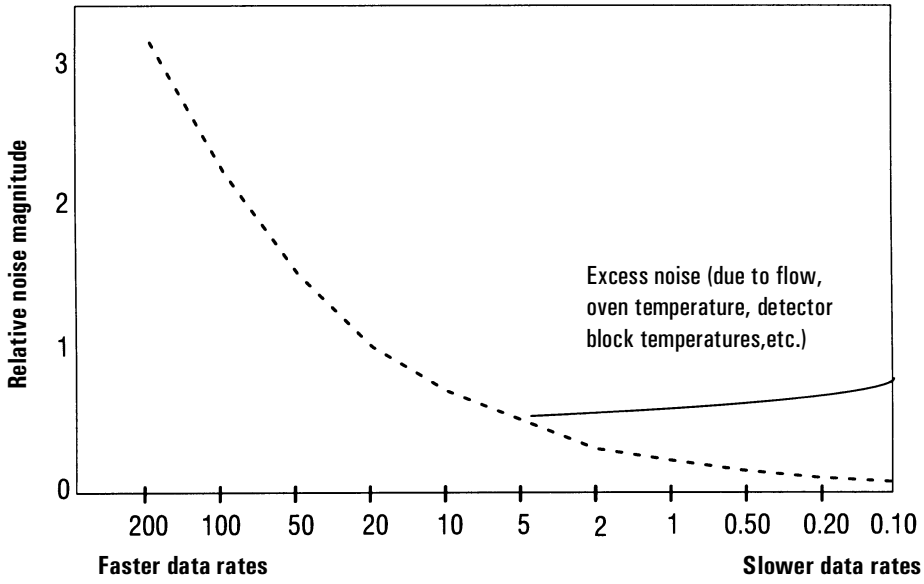


Figure 23 Relationship of noise to data rates

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 24 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]).

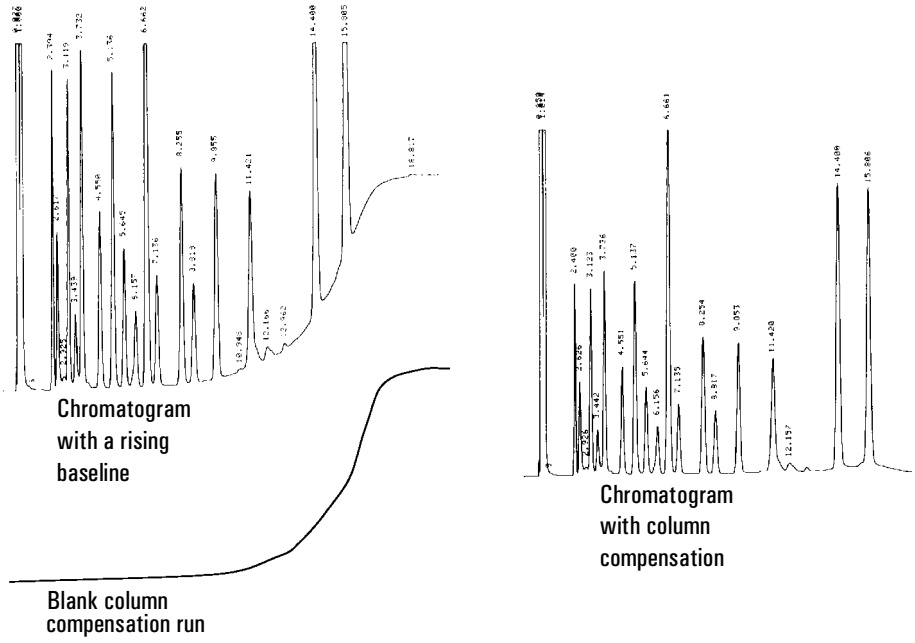
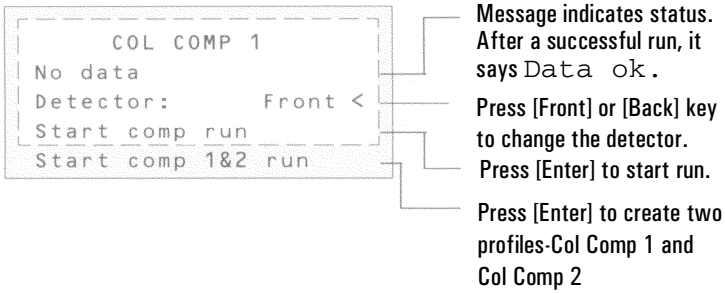


Figure 24 Column compensation

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.

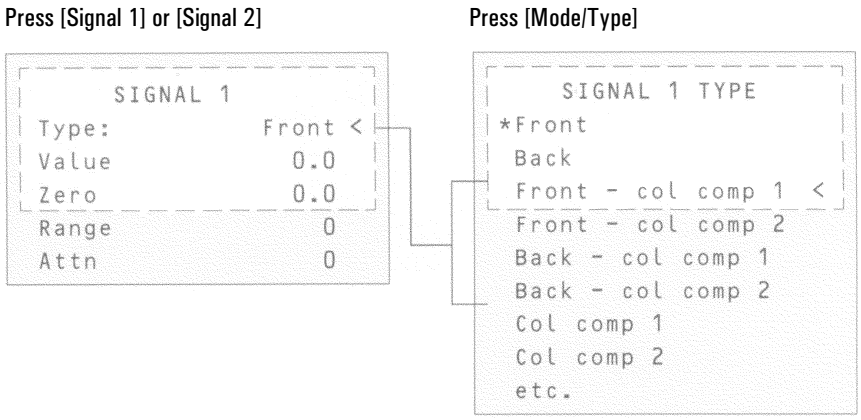


5. Select `Start comp run` or `Start comp 1&2 run`. Press [Enter].
   a. `Start comp run` creates one profile.
   b. `Start comp 1&2 run` creates two profiles (using different detectors and columns but the same oven temperature program).
6. If the run is successful, the first line of the control table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

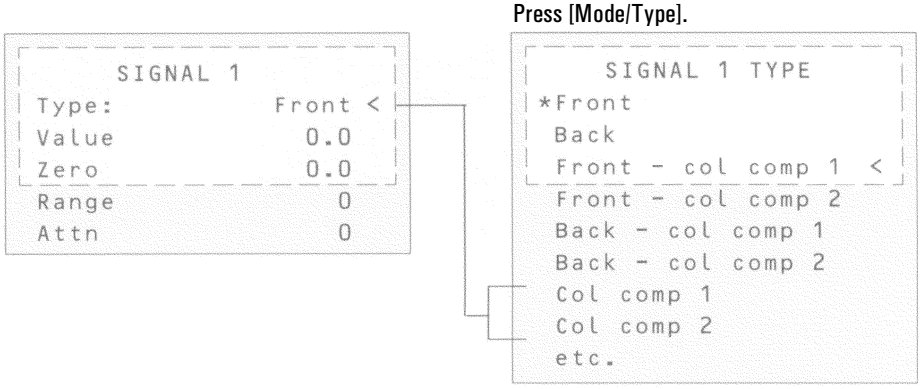
3. Scroll to `Type`: and press [Mode/Type].



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

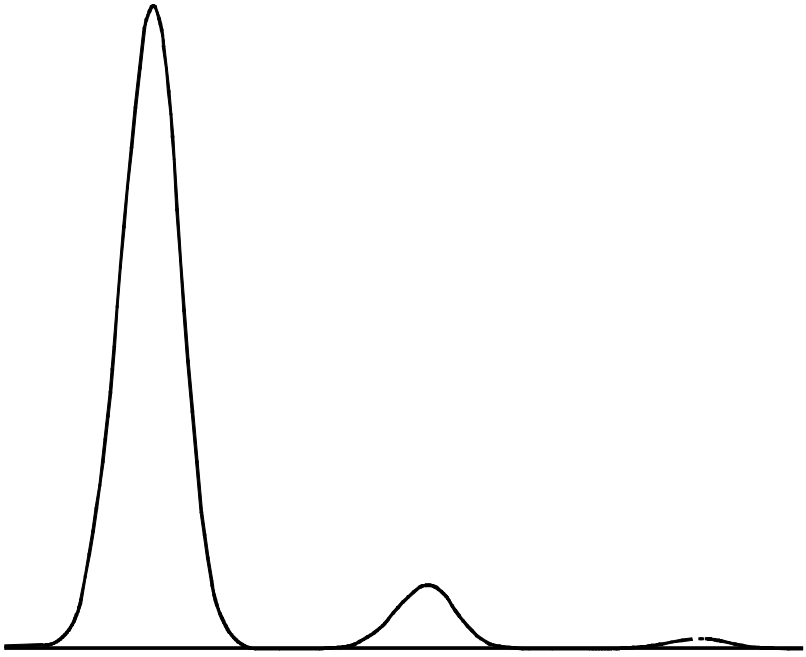
1. Press [Signal 1] or [Signal 2] to open the signal control table.
2. Scroll to `Type:` and press [Mode/Type].
3. Choose `Col comp 1` or `Col comp 2.`
4. Press [Start].



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.

7 Instrument Automation

Executing events during the run

Run time programming

Using run time events

Procedure: Programming run time events

The run table

Procedure: Adding events to the run table

Procedure: Editing events in the run table

Procedure: Deleting run time events

Clock time programming

Using clock time events

Procedure: Programming clock time events

Procedure: Adding events to the clock table

Procedure: Editing clock time events

Procedure: Deleting clock time events

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 "Valve Control".

Using run time events

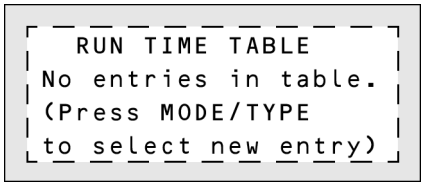
The [Run Table] key is used to program timed events.

You can control the following events during a run.

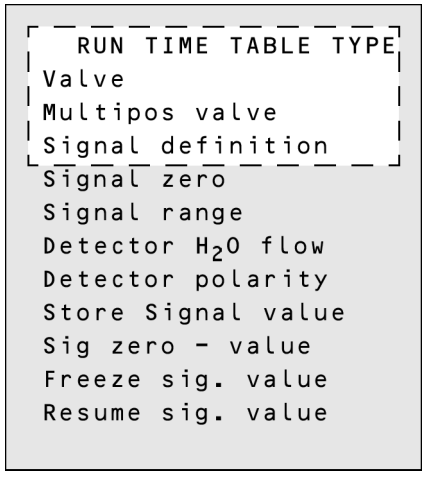
- Valves (1-8)
- Multiposition valve
- Signal type (see page 168)
- Analog signal zero, attenuation, and range
- Digital signal zero and baseline level shifts (see page 175)
- Auxiliary pressures (3, 4, 5)
- TCD negative polarity (on/off)
- NPD H_2 flow (on/off)
- Pausing (freezing) and resuming a signal 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.

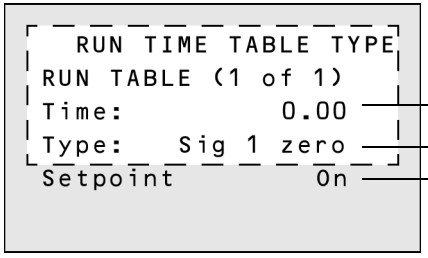


2. Press [Mode/Type] to see the run time event types.



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

3. Scroll to the event type to be programmed.



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:

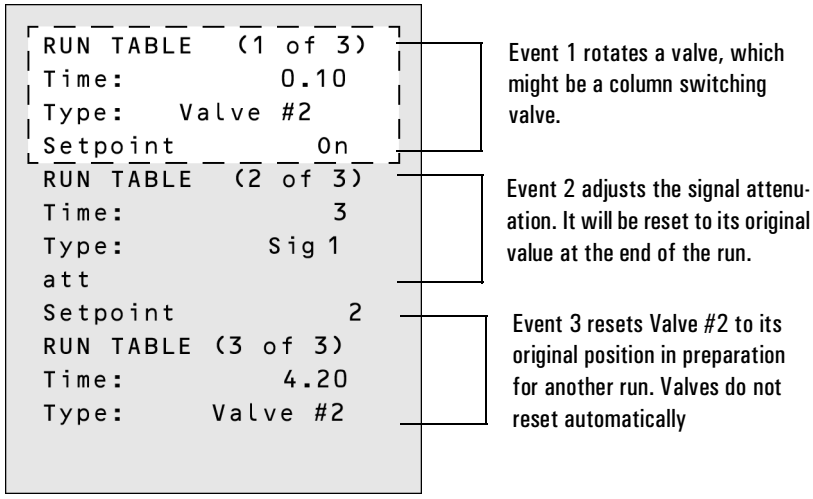


Figure 25 A run table example

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.

```
RUN TIME TABLE
Delete this event?
ENTER to delete
CLEAR to cancel
```

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

```
RUN TIME TABLE
Delete entire table?
ENTER to delete
CLEAR to cancel
```

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

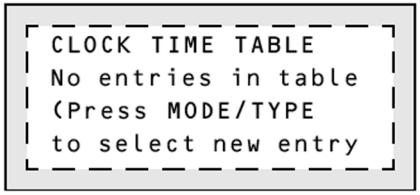
Using clock time events

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.

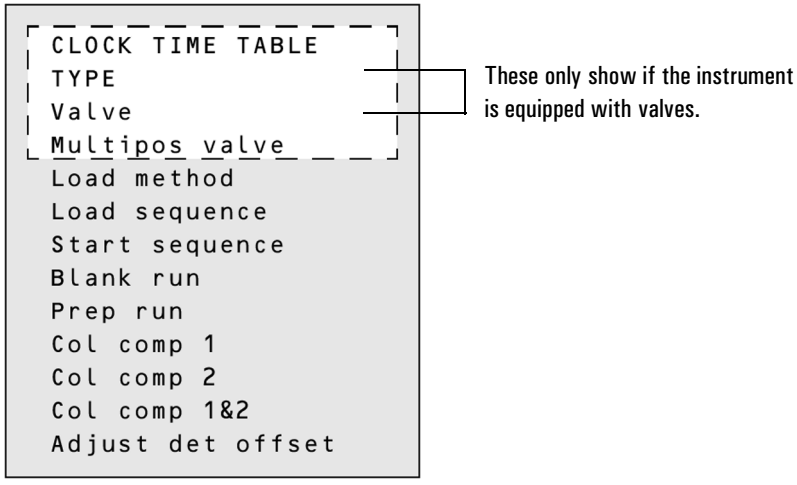
For example, the clock table could be used to start an analysis before you even get to work in the morning.

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.



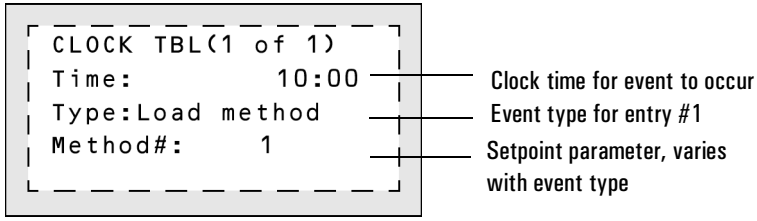
2. Press `[Mode/Type]` to view the clock time program types.



3. Scroll to select the parameter to be programmed.

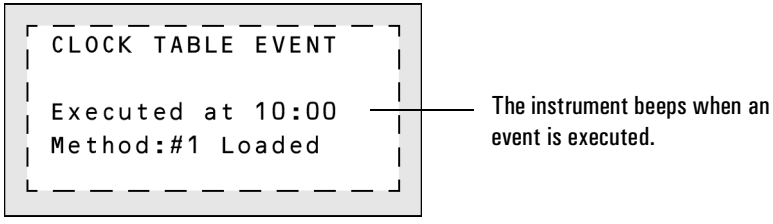
   For example, if the option “Load Method” is chosen for clock time event #1, the display would look similar to the one below.

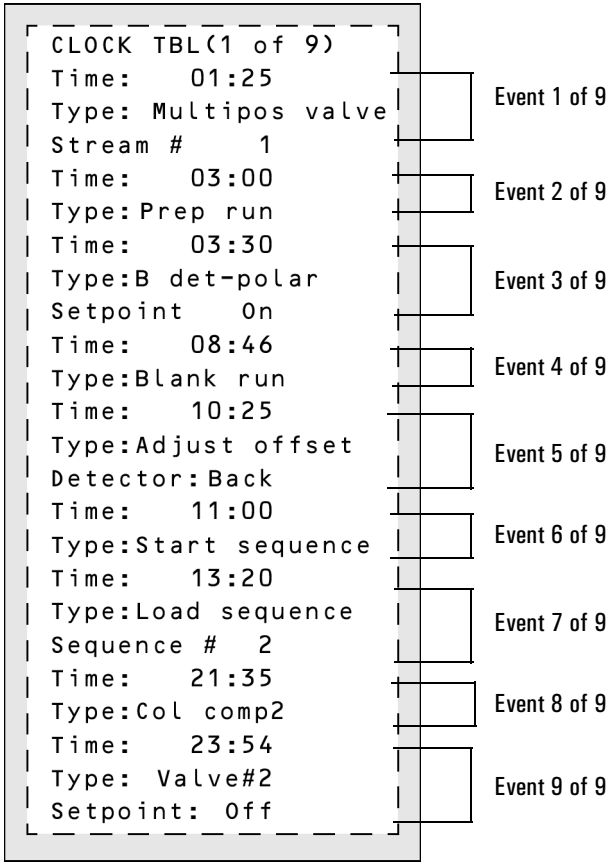
4. Edit `Time`: and `Method#`: setpoints for this event.



   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 26 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.

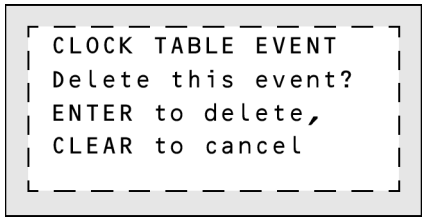
Repeat this process until all entries are added.

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.

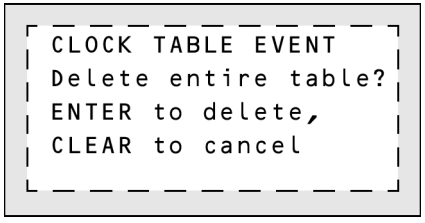
Procedure: Deleting clock time events

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

What is a method?

What can you do with it?

Creating a method

Procedure: Storing a method

Procedure: Loading a previously stored method

Procedure: Loading the default method

Method mismatch

User-entered configuration changes

Hardware configuration changes

Procedure: Modifying a previously stored method

Procedure: Deleting a stored method

Method listings

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:

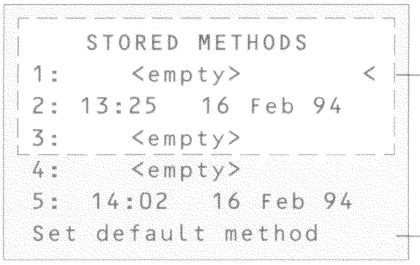
- The active method—the setup that you are presently using.
- Stored methods—one of the five methods that can be stored in the GC.
- The default method—a set of default parameters for the GC. It can be reloaded at any time.

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:

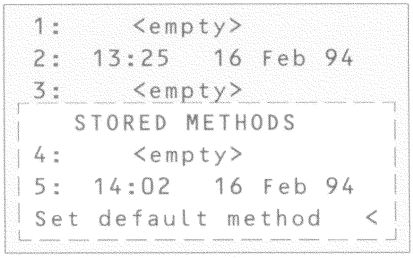
- Oven
- Front/Back inlet
- Column 1 & 2
- Front/Back detector
- Signals 1 & 2
- Aux #1-5
- Post run
- Valve # 1-8
- Run time table
- Front and back injectors
- Sample tray

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.

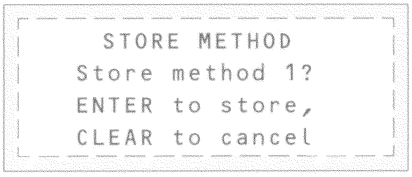
Procedure: Storing a method

To store a method:

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

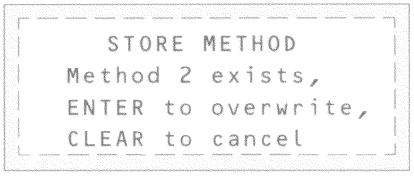


2. Press the [Store] key. You are then asked to confirm the store.



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:

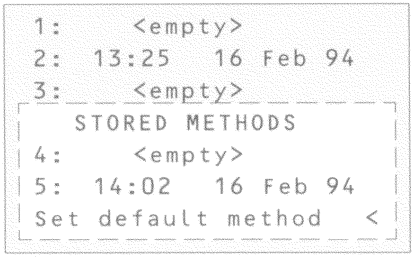


   - [Enter] to replace the existing method with the new one and return to the STORED METHODS status table.
   - [Clear] to return to the STORED METHODS status table without storing the method.

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].

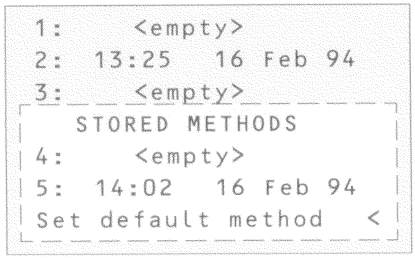
4. Press [Enter] to load the method. The selected method replaces the active method.

   [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 "The Keyboard and Display".

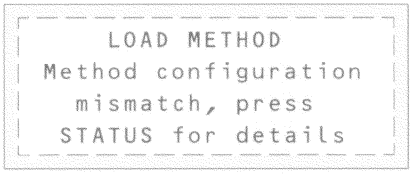
Method mismatch

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.

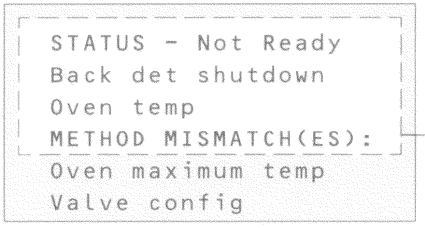
Mismatches are caused by user changes (different choice of carrier gas, etc.) or by hardware changes (replace a TCD with an FID, etc.) that are made after the method is stored.

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 a μ-ECD. If you now load a method that uses the FID, the FID setpoints cannot load. They will be ignored and current μ-ECD setpoints will be retained. All other setpoints that can be loaded, will be loaded.

Procedure: Modifying a previously stored method

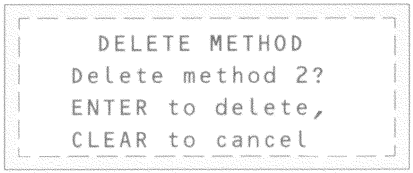
When a method is loaded it replaces the active method.

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

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 The Automatic Sampler

Injector control table
Procedure: Editing injector set-points

Configuring the injector
Procedure: Configuring the injector

Sample tray setpoints
Procedure: Editing the sample tray setpoints
Procedure: Configuring the bar code reader

Storing injector setpoints

The Automatic Liquid Sampler

This section describes how to configure and use your 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 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 automatic liquid sampler, refer to its Operation Guide (Part no. G2612-90117).

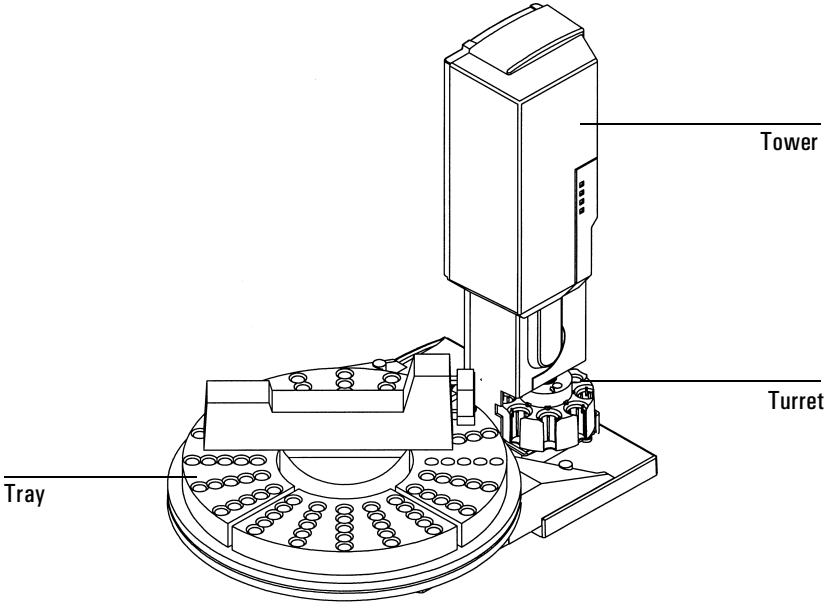
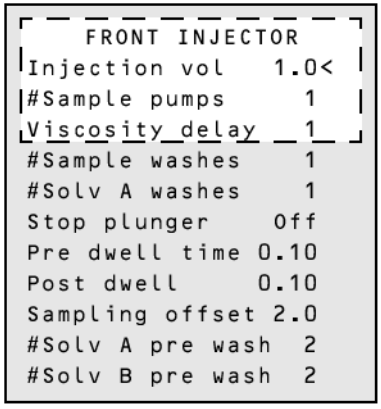


Figure 27 Automatic sampler components

Injector control table

Press [Front Injector] or [Back Injector].



`Injection volume`—Sample volume to be injected. Press [Mode/Type] to select. The available volumes depend on the syringe size configured. See "Configuring the injector" on page 209.

- The selections represent 2%, 10%, 20%, 30%, 40%, and 50% of syringe size
- Turn the injection volume Off to disable the injector

`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.
    - Press [Mode/Type] to make a selection for syringe size.

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 mode

The turret waste bottle positions are controlled using the [Mode/Type] key:

- `Use both A and B` alternates between the two waste bottles
- `Use only A bottle` uses bottle A only
- `Use only B bottle` uses bottle B only

Use B2 wash

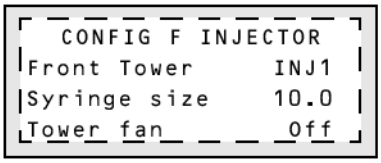
You can enable this option to use two 4 mL vials of solvent B, increasing the number of runs you can make before refilling solvent vials.

- 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 mode* 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 an injector with an eight sample vial turret

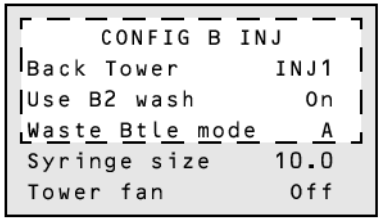
1. Press [Config][Front Injector] or [Config][Back Injector].



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 `Syringe size`, in µL.
4. With the cursor on Tower Fan, use the [On] or [Off] key to select fan usage.
   - In general, leave the fan On.

Procedure: Configuring an injector with a three sample vial turret

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



2. Set the B2 wash bottle usage On or Off.
3. To select the waste bottle mode, press [Mode/Type].
4. Enter the installed syringe size, in µL, for `Syringe size`.
5. With the cursor on Tower Fan, use the [On] or [Off] key to select fan usage.
   - In general, leave the fan On.

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

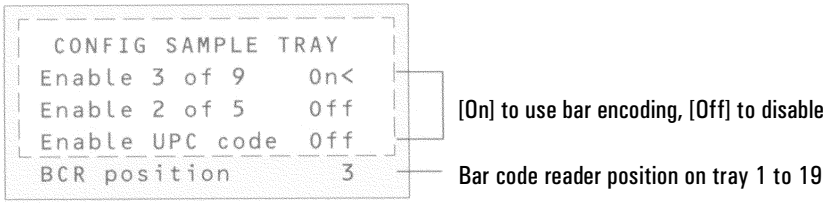
1. Press [Sample tray] to access the sample tray and bar code reader setpoints.

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 "Procedure: Storing a method". This method becomes a part of the sequence used to run the samples. For more information on injector sequences and sequence control, see "Analytical Sequences".

10 Valve Control

The valve box
Heating the valves
Valve temperature programming
Configuring an Aux thermal zone

Valve control
The valve drivers

The internal valve drivers

The external valve drivers

Valve configurations
Procedure: Configuring a valve

Valve control
Procedure: Controlling valves from the keyboard
From the run or clock time tables

Valve control examples
Simple valve—column selection
Gas sampling valve
Multiposition stream selection valve and sampling valve

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

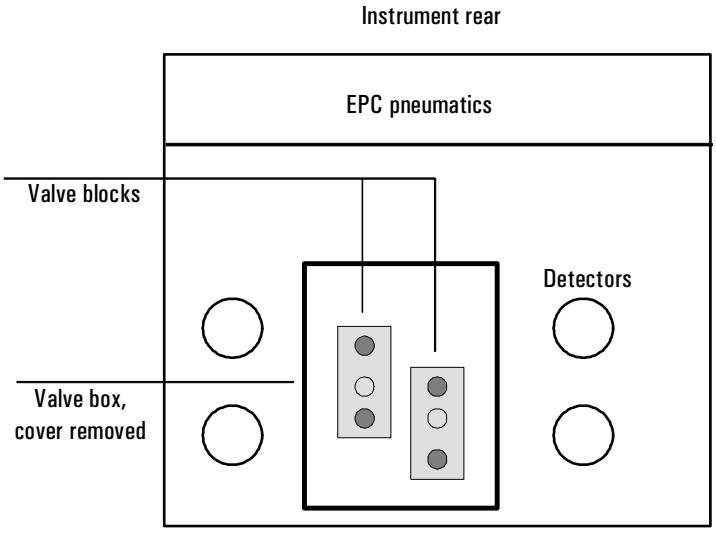


Figure 28 The valve box

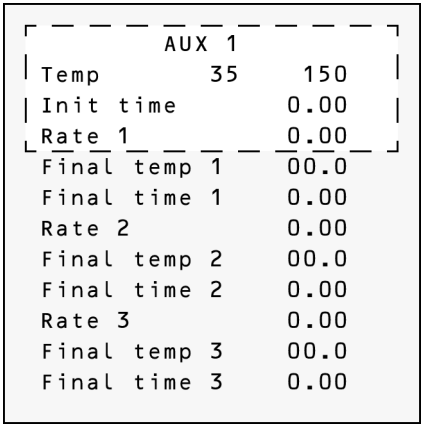
Heating the valves

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.

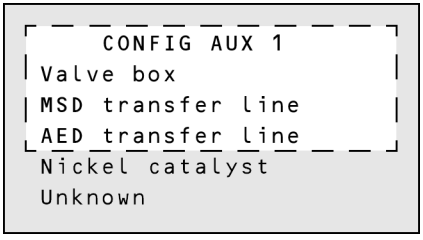
Valve temperature programming

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 107 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.

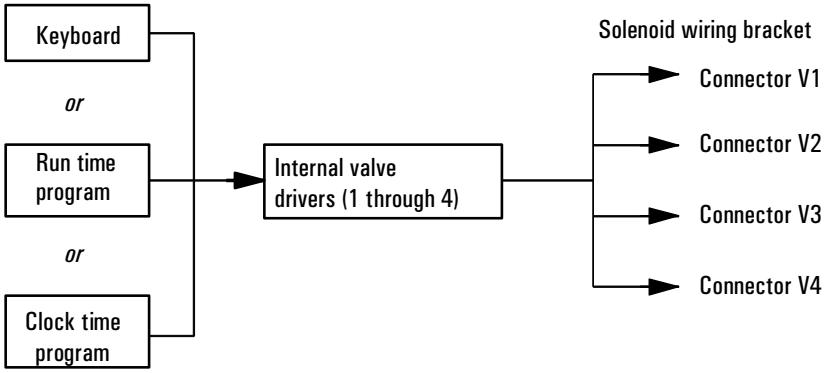


Figure 29 Internal valve drivers

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 216.

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.

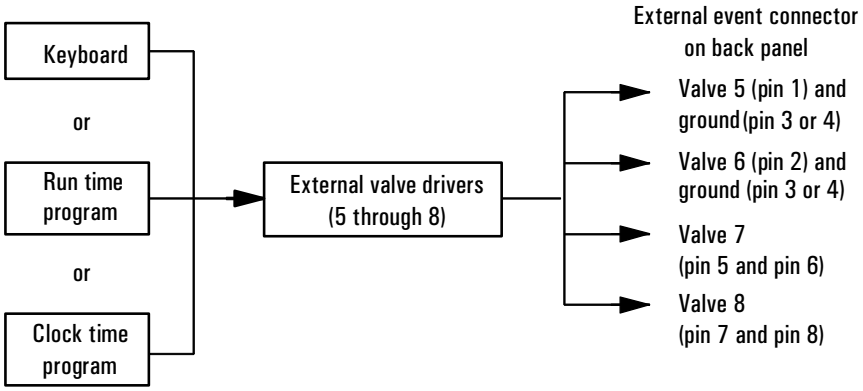


Figure 30 External valve drivers

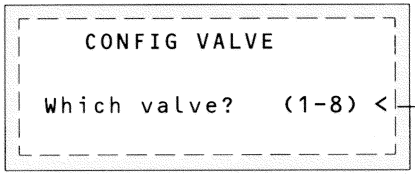
Valve configurations

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 222.
- 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 221.
- 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 223.
- Other—could be anything.
- Not installed—self-explanatory.

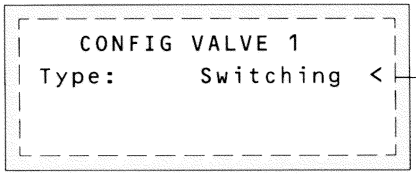
Procedure: Configuring a valve

1. Press [Config] [Valve #].

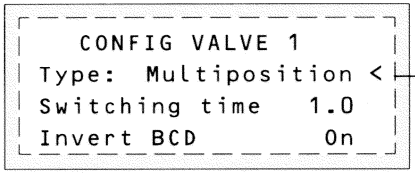


2. Specify the valve driver to be configured. Type the number (1 in this example) and press [Enter].

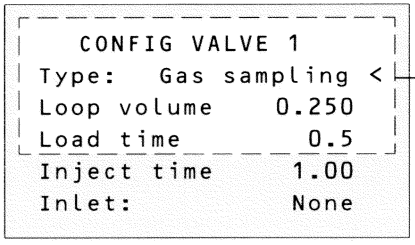
3. The present type for this driver is shown.



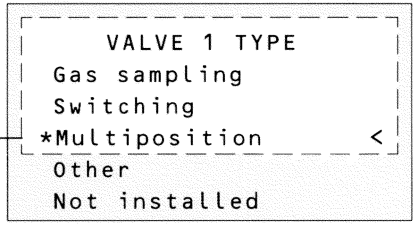
or



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 "Procedure: Programming run time events". See "Procedure: Programming clock time events".

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.

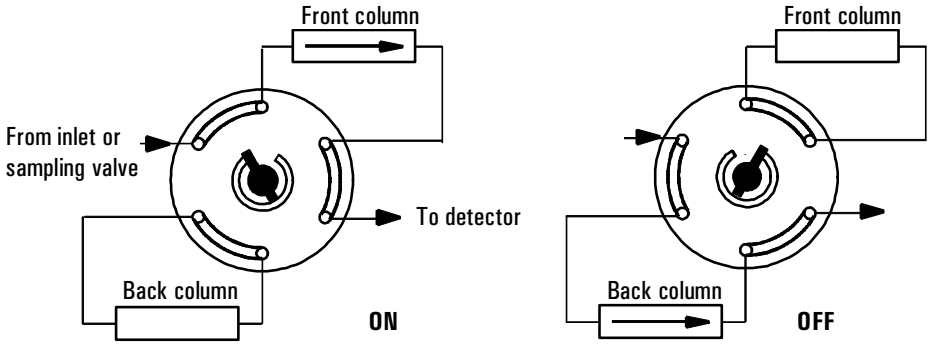


Figure 31 A column selection valve

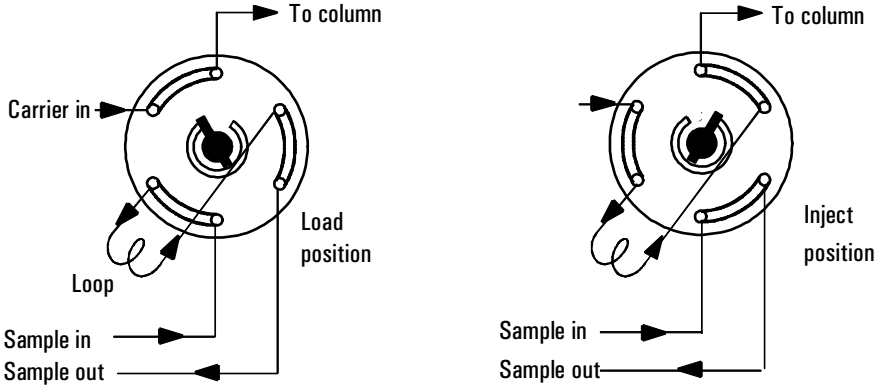
The column is selected by pressing [Valve #] <scroll to valve 2> [On] (for the front column) or [Off] (for the back column). This run table ensures that the valve is in the Off state between runs:

```
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

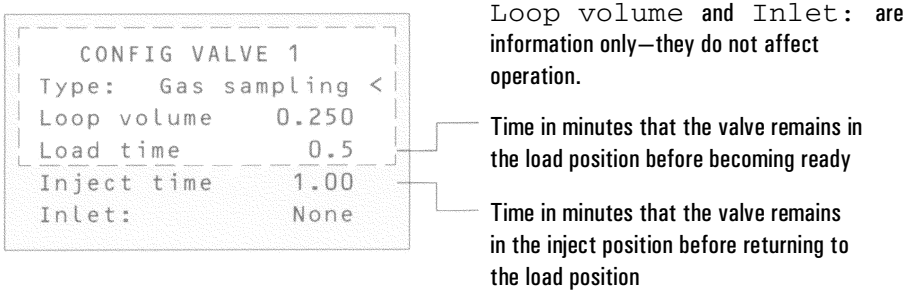
If a valve is configured as a gas sampling valve, it starts a run automatically when it is switched to the Inject position. This can be done with a keyboard command or by a subsequence or clock table entry. You may have two gas sampling valves installed.



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 32 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.



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 216 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.

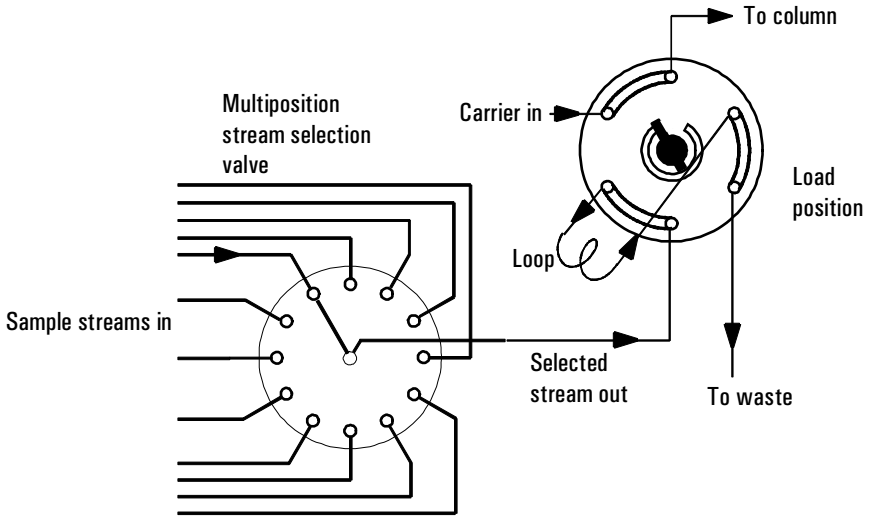
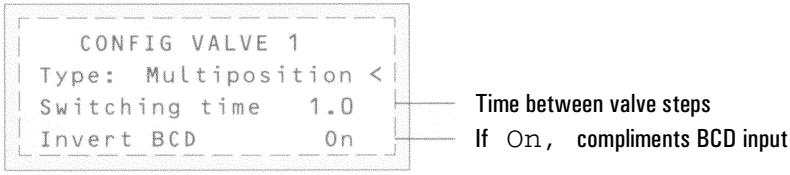


Figure 33 Multiposition valve with gas sampling valve

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 33. 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 "Procedure: Creating a valve subsequence".

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.

11 Analytical Sequences

What is a sequence?

What can you do with it?

Defining a sequence

- Priority sequence
- Subsequences
- Post Sequence

Procedure: Creating a sequence

- Procedure: Creating a sampler subsequence
- Procedure: Creating a valve subsequence
- Procedure: Setting the Post Sequence events

Procedure: Storing a sequence

Procedure: Loading a previously stored sequence

Procedure: Modifying a previously stored sequence

Procedure: Deleting a sequence

Sequence control

- Sequence status
- Procedure: Starting/running a sequence
- Procedure: Pausing and resuming a sequence
- Procedure: Stopping a sequence
- Aborting a sequence

Special considerations when using an integrator

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 34, 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 35.

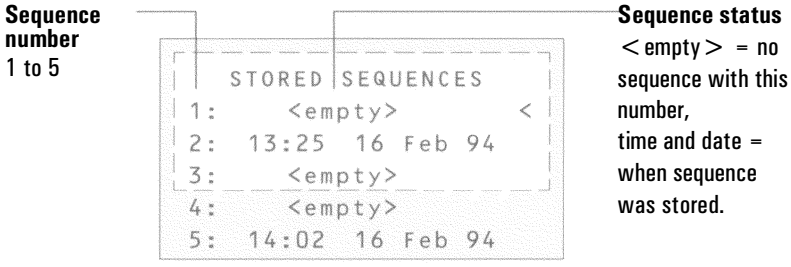


Figure 34 Stored sequence control table

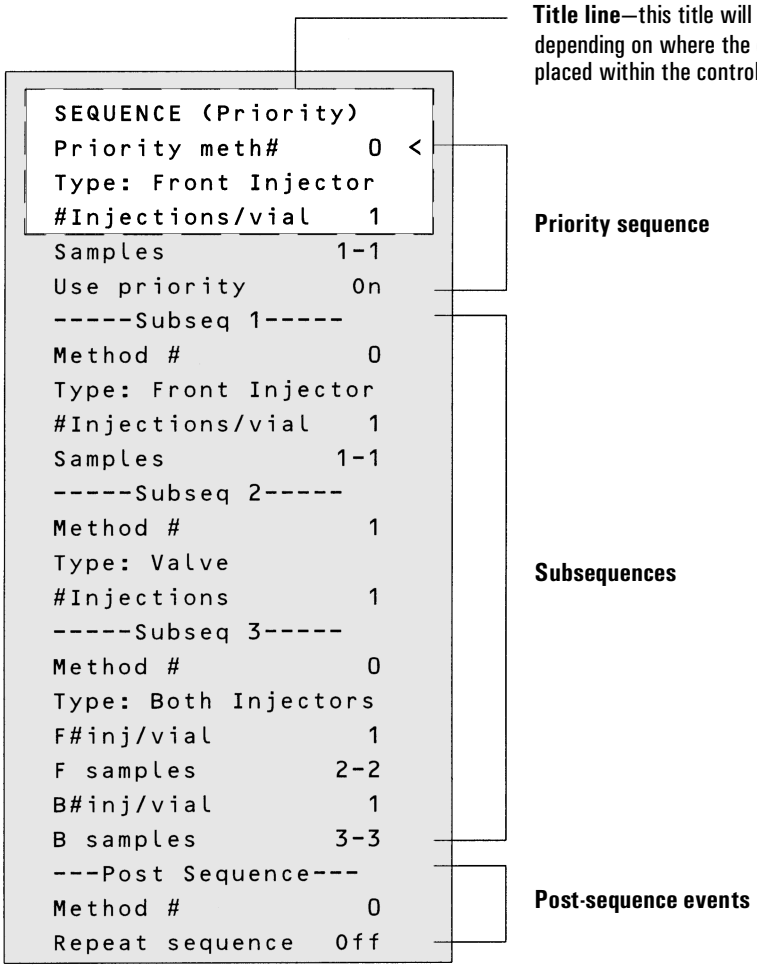


Figure 35 Sequence definition control table

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):

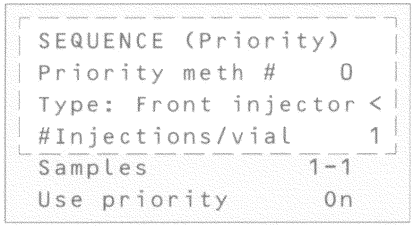
- Priority sequence—this is a special provision that allows you to interrupt a sequence to analyze urgent samples.
- Subsequences—each subsequence consists of the number of a stored method and information that defines a set of samples and calibrators to be analyzed using the method. There may be up to 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



Valve

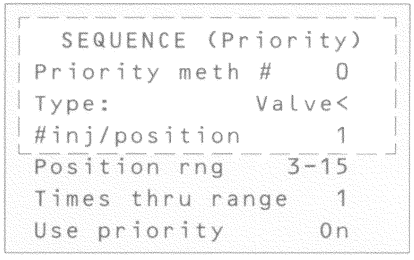


Figure 36 Priority sequences

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

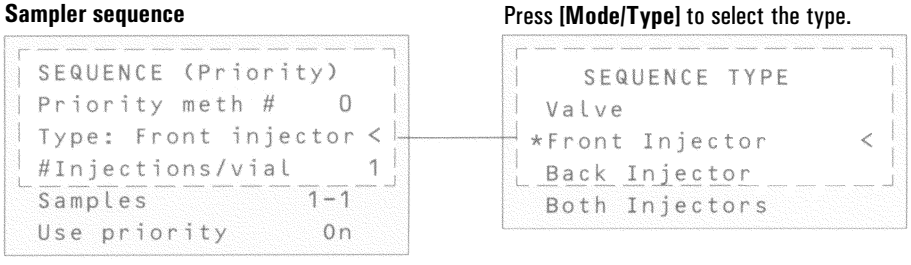
1. Press [Seq] to open the sequence control table.
2. Create a Priority Sequence, if desired. This is either a valve or sampler subsequence, with two differences. The method line is labeled `Priority meth #`. An additional line, labeled `Use priority`, may be set either `On` or `Off`.
3. Create one to five subsequences. Subsequences may be either sampler subsequences (below) or valve subsequences (page 231). 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`.

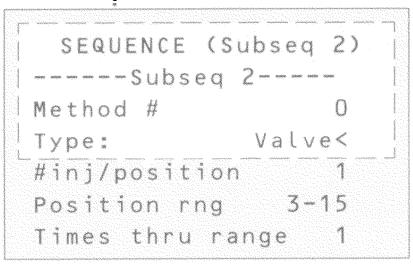
Procedure: Creating a valve subsequence

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

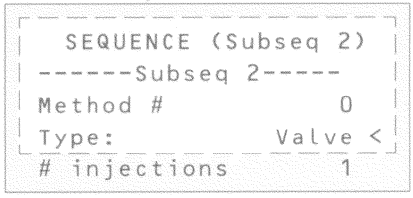


Figure 37 Valve subsequences

5. Enter the valve sequence parameters (the first three appear only if a multiposition 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

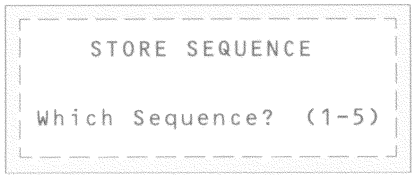
1. Scroll to the `Post Sequence` title section.

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.

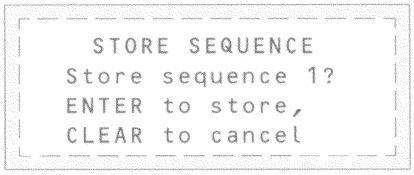
3. `Repeat sequence`—`On` keeps repeating the sequence. This function is useful for valve sequences. `Off` halts the sequence at the end. Turn `Repeat sequence` On or Off.

Procedure: Storing a sequence

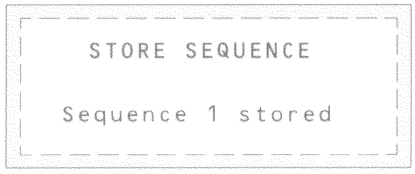
1. Press [Store][Seq] to open the Store Sequence control table.



2. Enter an identifying number for the sequence.



3. Press [Enter] to store the sequence.



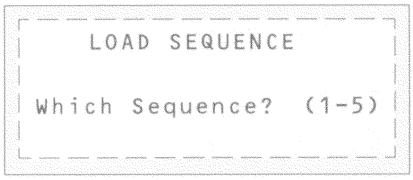
If the sequence number you specified already exists, you will be prompted to either:

- 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.

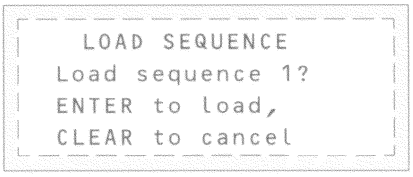
Sequences can also be stored from within the `STORED SEQUENCES` status setpoint table by scrolling the cursor to the appropriate sequence number and pressing the [Store] key.

Procedure: Loading a previously stored sequence

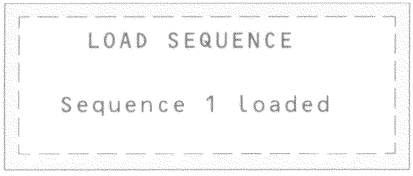
1. Sequences can be loaded by pressing [Load][Seq].



2. Press a number key to select the sequence to be loaded.



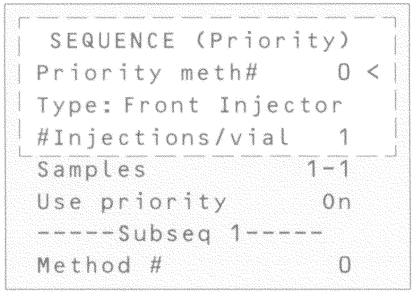
3. Press [Enter] to load the sequence or cancel this by pressing [Clear].
4. If you press [Enter], the load is confirmed. This is now the active sequence.



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

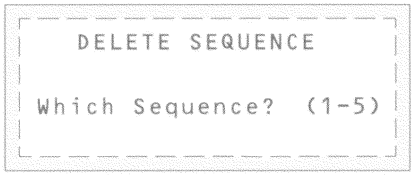
1. Load the sequence you wish to edit.
2. Open the sequence control table. Scroll to the parameter within the subsequence or Post Sequence you wish to edit.



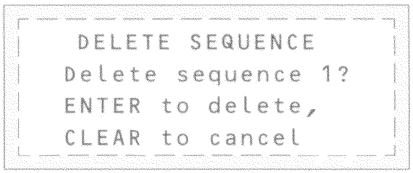
3. Make the changes.
4. To save the new values, store the sequence under its original number.

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:

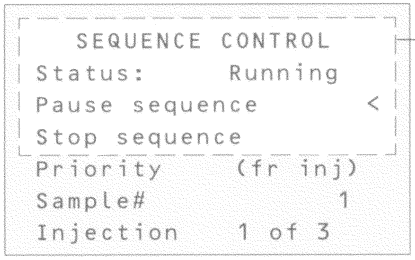
Sequence control

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:

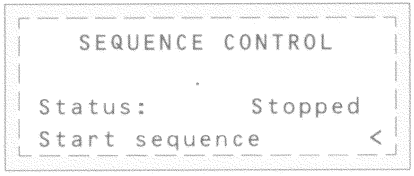
- Start/running
- Ready wait
- Paused/resume
- Stopped
- Aborted
- No sequence



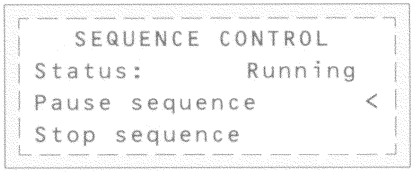
Sequence Control—diplays the current status of the sequence, shows which subsequence is currently active, current sample # or valve position, and which injection number of multiple runs is currently executing.

Procedure: Starting/running a sequence

To start a sequence, scroll to the `Start sequence` line and press [Enter].



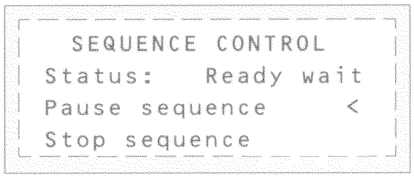
Pressing [Enter] changes the sequence status to `Running`.



The sequence continues to run until all subsequences are executed, or until one of the events described on page 239 occurs.

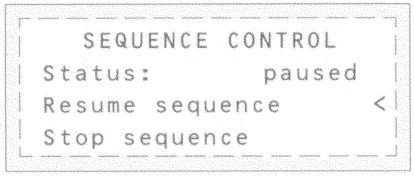
Ready wait

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.

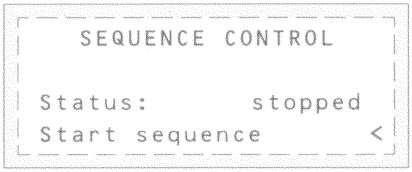
2. To continue the paused sequence, scroll to `Resume sequence` and press [Enter].

   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.

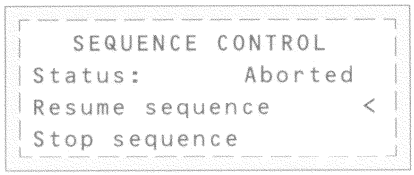


A sequence stops at the end of the last active subsequence unless `Repeat sequence` is `On` in the Post Sequence events.

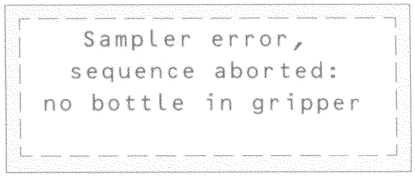
Aborting a sequence

When a sequence is aborted, it stops immediately without waiting for the current run to finish. These will cause a sequence to abort:

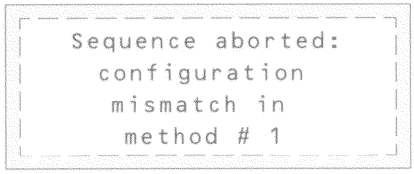
A run is stopped by pressing the [Stop] key.



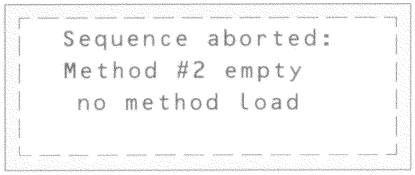
A sampler error occurs producing an error message.



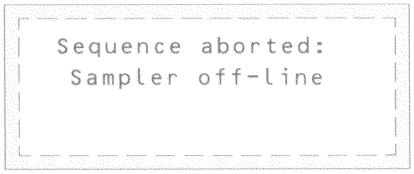
The GC detects a configuration mismatch during a method load



A running sequence tries to load an empty method.



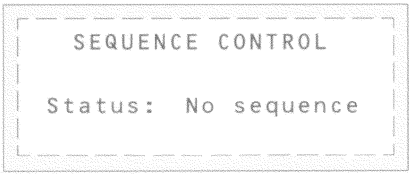
The sampler is turned off.



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].
- 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

Not Ready messages

Temperature zone not ready

Pressure and/or flow not ready

Detector not ready

Valve not ready

Other not ready messages

Shutdown messages

Warning messages

Fault messages

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* sections.

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 23 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 23 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.
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.

Not Ready messages

A listing of the Not Ready messages is given in Table 24 below.

Table 24 Not Ready Messages

Status message	Run log entry	Comments
Temperature zone not ready messages		
Oven temp	Not ready: Oven temp ####	See page 248.
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 249.
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: F inl pres	
Front inlet flow	Not ready: F inlet flow ##.#	
Back inlet pressure	Not ready:	
Back inlet flow	Not ready: B inlet flow ##.#	See page 249.
Front det H2 flow	Not ready: F det H2 flow	
Front det gas 2	Not ready: F det gas 2	
F det makeup gas	Not ready: F det makeup	

Table 24, continued

Status message	Run log entry	Comments
Back det H2 flow	Not ready: B det gas 2	
Back det gas 2	Not ready: B det gas 2	
B det makeup gas	Not ready: B det makeup	
Aux 3 pressure	Not ready:	See page 249.
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 249.
Back det waiting	Not ready: Back det on wait	See page 249.
Front det igniting	Not ready: Front det ignite	See page 250.
Back det igniting	Not ready: Back det ignite	See page 250.
Front det adjusting	Not ready: Front det adjust	See page 250.
Back det adjusting	Not ready: Back det adjust	See page 250.
Front det equib time	Not ready: Front det equib	See page 250.
Back det equib time	Not ready: Back det equib time	See page 250.
Front det shutdown	Not ready: Front det shutdown	See page 250.
Back det shutdown	Not ready: Back det shutdown	See page 250.
F NPDBead slewing	Not ready: Front NPD slewing	See page 251.
F NPDBead slewing	Not ready: Back NPD slewing	See page 251.

Table 24, 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 252.
Back inlet purging	Not ready: B inlet purge	Inlet in split mode is purging. Press [Prep Run]. See page 252.
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 251.
Multiposition valve	Not ready: Multiposition valve	See page 251.
Gas sampling valve 1	Not ready: Gas sampling valve 1	See page 251.
Gas sampling valve 2	Not ready: Gas sampling valve 2	See page 251.

Table 24, continued

Status message	Run log entry	Comments
Other not ready messages		
Diagnostics mode	Not ready: Diagnostics active	See page 252.
Test in progress	Not ready: Test in progress	A diagnostic test is in progress. Wait until it is completed.
Front inj door open	Not ready: Front inj door open	
Back inj door open	Not ready: Back inj door open	
Host system	Not ready: Host system	See page 252.
External device		An device connected to the Remote Start connector is not ready.
Power on in progress	Power-on restart: Blank run	See page 253.

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 "The Flame Ionization Detector" or "The Flame Photometric Detector".

Front det adjusting
Back det adjusting

The GC is not ready because the NPD or µ-ECD is adjusting its baseline to reach the offset (FID) or output (µ-ECD) setpoint. The µ-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.

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 25 Shutdown Messages

Shutdown no.	Popup message	Comments
1	Oven shut off	See page 255.
2	Oven cryo shutdown	See page 255.
3	Front inlet pressure shutdown	See page 256.
4	Front inlet flow shutdown	See page 256.
5	Back inlet pressure shutdown	See page 256.
6	Back inlet flow shutdown	See page 256.
7	Front detector fuel gas shutdown	See page 256.
8	Front detector air/ref shutdown	See page 256.
9	Front detector makeup shutdown	See page 256.
10	Back detector fuel gas shutdown	See page 256.
11	Back detector air/ref shutdown	See page 257.
12	Back detector makeup shutdown	See page 257.
13	Pres aux 3 shutdown	See page 257.
14	Pres aux 4 shutdown	See page 257.
15	Pres aux 5 shutdown	See page 257.
16	Multiposition valve is not switching	See page 257.
17	Can't reach setpoint of multipos valve	See page 257.
18	Front inlet cryo shutdown	See page 258.
19	Back inlet cryo shutdown	See page 258.
20	Aux 1 cryo shutdown	See page 258.
21	Aux 2 cryo shutdown	See page 258.
22	Front inlet heating too slowly: temperature shut off	See page 259.
23	Back inlet heating too slowly: temperature shut off	See page 259.

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 timeout 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 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 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 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 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 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 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.

Warning messages

Table 26 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 26 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 263.
104	Sig 2 buffer full	Sig 2 buffer full	Possible data loss: Sig 2 buffer full	See page 263.
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 26, 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 26, 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		For Volatiles interface, a setpoint conflicted with the Sampling End time parameter. Check your method. See "The Volatiles Interface" for more information.
133		Chgd Col 2 Init time to ###.## ; avoids Sampling End problem		
138	F inj/inlet mismatch	Front injector incompatible with front inlet		
138	b inj/inlet mismatch	Back injector incompatible with front inlet		
140		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 Volatiles Interface" for more information.
141		Chgd BI Saver time to ###.## ; avoids Sampling End problem		
142		Chgd FI Purge time to ###.## ; avoids Sampling End problem		
142		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 Agilent Cerity or ChemStation software) 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 Cerity or 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 Cerity or ChemStation software 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.

Fault messages

Table 27 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 27 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 270.
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 27, 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 271.
215	Back det flame out	Back detector flame out	Not ready: Back det flame out	See page 271.
216	F TCD filament open	Front TCD filament open	Not ready: F TCD filament open	See page 271.
217	B TCD filament open	Back TCD filament open	Not ready: B TCD filament open	See page 271.
218	F TCD filament short	Front TCD filament shorted	Not ready: F TCD filament short	See page 272.
219	B TCD filament short	Back TCD filament shorted	Not ready: B TCD filament short	See page 272.
220	Heater overcurrent	Heater overcurrent. Thermal shutdown.		
221	Thermal shutdown		Not ready:	See page 272.
222	Oven temp too hot	Oven thermal shutdown	Not ready: Thermal shutdown	See page 272.
223	Oven temp too cool	Oven thermal shutdown	Not ready: Thermal shutdown	See page 272.
224	Oven temp sensor	Oven thermal shutdown	Not ready: Thermal shutdown	See page 272.
225	F det temp too hot	Front detector thermal shutdown	Not ready: Thermal shutdown	See page 272.

Table 27, 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 272.
227	B det temp too hot	Back detector thermal shutdown	Not ready: Thermal shutdown	See page 272.
228	B det temp sensor	Back detector thermal shutdown	Not ready: Thermal shutdown	See page 272.
229	F inl temp too hot	Front inlet thermal shutdown	Not ready: Thermal shutdown	See page 272.
230	F inl temp sensor	Front inlet thermal shutdown	Not ready: Thermal shutdown	See page 272.
231	B inl temp too hot	Back inlet thermal shutdown	Not ready: Thermal shutdown	See page 272.
232	B inl temp sensor	Back inlet thermal shutdown	Not ready: Thermal shutdown	See page 272.
233	Aux I temp too hot	Aux 1 thermal shutdown	Not ready: Thermal shutdown	See page 272.
234	Aux I temp sensor	Aux 1 thermal shutdown	Not ready: Thermal shutdown	See page 272.
235	Aux 2 temp too hot	Aux 2 thermal shutdown	Not ready: Thermal shutdown	See page 272.
236	Aux 2 temp sensor	Aux 2 thermal shutdown	Not ready: Thermal shutdown	See page 272.
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 27, 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 27, 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	
262	RS232 defective	Host communications: RS232 defective	Not ready: RS232 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 27, 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	

Table 27, continued

Fault no.	Status message	Popup message	Run log	Comments
290	Zones not updating	Zones not updating	Not ready: Zones not updating	
293	Zone heater driver	Zone heater driver	Not ready: Zone heater driver	

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 Flame Ionization Detector", "The Flame Photometric Detector" 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.

13 Introduction to Inlets

Inlet types

Using hydrogen

Procedure: Pressure units: Select psi, kPa, bar

The inlet and column control tables

The column control tables

The column control table—defined capillary columns

The column control table—packed or undefined capillary columns

What is gas saver?

Procedure: Using gas saver

Pre Run and Prep Run

The [Prep Run] key

Procedure: Auto Prep Run

Septum purge

Introduction to Inlets

Inlet types

The 6890 GC has five types of inlets available. All are offered with electronic pneumatics control (EPC) and two are offered without.

Table 28 Inlet Types

Inlet type	Gas control
Split/splitless	EPC and nonEPC
Purged packed	EPC and nonEPC
Cool on-column	EPC only
Programmed temperature vaporization	EPC only
Volatiles interface	EPC only

Using hydrogen

WARNING When using hydrogen (H_2), as the carrier gas, be aware that hydrogen (H_2) gas can flow into the oven and create an explosion hazard. Therefore, be sure that the supply is off until all connections are made, and ensure that the inlet and detector column fittings are either connected to a column or capped at all times when hydrogen (H_2) gas is supplied to the instrument.

WARNING Hydrogen (H_2) is flammable. Leaks, when confined in an enclosed space, may create a fire or explosion hazard. In any application using hydrogen (H_2), leak test all connections, lines, and valves before operating the instrument. Always turn off the hydrogen (H_2) supply at its source before working on the instrument.

Table 29 An Overview of Inlets

Inlet	Column	Mode	Sample concentration	Comments	Sample to column
Split/splitless	Capillary	Split	High		Very little
		Pulsed split	High	May be useful with large injections	Very little
		Splitless	Low		All
		Pulsed splitless	Low	Useful with large injections	All
Cool on-column	Capillary	n/a	Low or labile	Minimal discrimination and decomposition	All
Purged packed	Packed	n/a	Any		All
	Large capillary	n/a	Any	OK if resolution not critical	All
Programmed temperature vaporization	Capillary	Split	High		Very little
		Pulsed split	High		Very little
		Splitless	Low		All
		Pulsed splitless	Low		All
		Solvent vent	Low	Multiple injections concentrate analytes and vent solvent	Most
Volatiles interface	Capillary	Direct	Low	Lowest dead volume	All
		Split	High	Max flow = 100 mL/min	Very little
		Splitless	Low		All

Table 30 Column Size and Carrier Gas Flow Rate

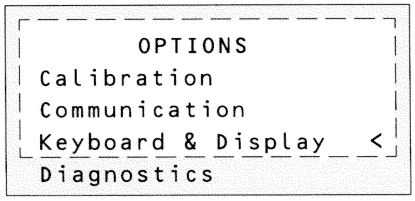
Column type	Column size	Carrier gas flow rate	
		Hydrogen	Helium
Packed	1/8-inch		30
	1/4-inch		60
Capillary	50 µm id	0.5	0.4
	100 µm id	1.0	0.8
	200 µm id	2.0	1.6
	250 µm id	2.5	2.0
	320 µm id	3.2	2.6
	530 µm id	5.3	4.2

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.

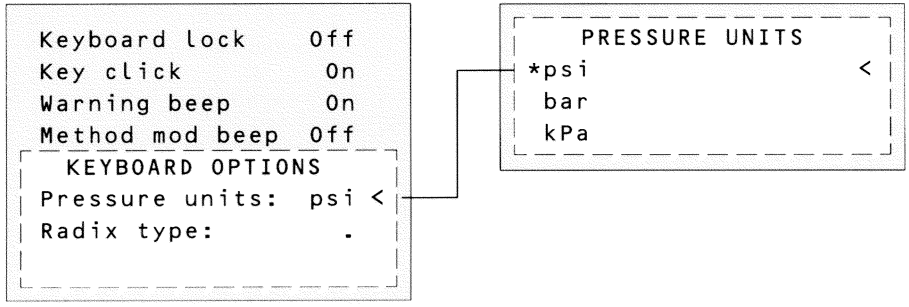
Procedure: Pressure units: Select psi, kPa, bar

You can display pressure in psi, bar, or kPa. To check the units you are using, pressing the [Info] key while the cursor is on the Pressure line of a control table. To change the display units:

1. Press [Options].
2. Scroll to `Keyboard & Display` and press [Enter].



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



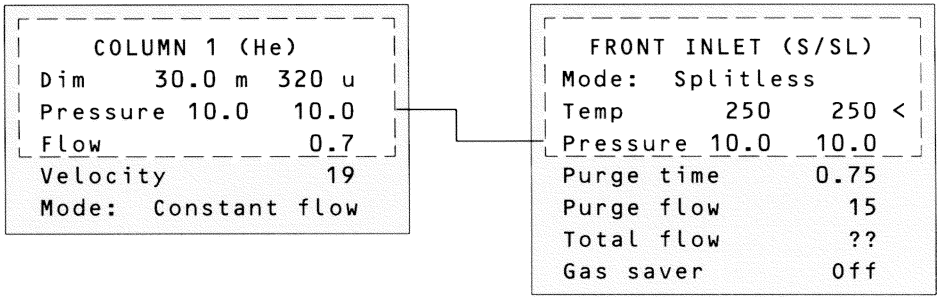
4. Choose a new pressure unit and press [Enter].

Table 31 Pressure Unit Conversions

To convert	to	Multiply by
psi	bar	0.0689476
	kPa	6.89476
bar	psi	14.5038
	kPa	100
kPa	psi	0.145038
	bar	0.01

The inlet and column control tables

The tables for the inlet and column are interrelated. If you set a pressure at the column control table, that same pressure setting is active on the inlet control table, and vice versa. Although pneumatics can be controlled from either the column or the inlet, the column should be considered first.



Note that the pressure readings—both setpoint and actual—are identical on the column and inlet control tables.

The column control tables

The control tables change depending on your column configuration. The next few pages describe the column control tables for the two types of columns, capillary and packed.

The column control table—defined capillary columns

If your column is defined, your control table will be similar to Figure 38.

The title This heading identifies the column—Column 1 or Column 2— and the type of carrier gas configured to the inlet (in parentheses).

`Dim` This line shows the column dimensions you have specified. Column length is in meters (`m`) and column inside diameter is in microns (µ).

Pressure, flow, and velocity are related. If the column is defined, enter any one of them and the GC computes and displays the other two.

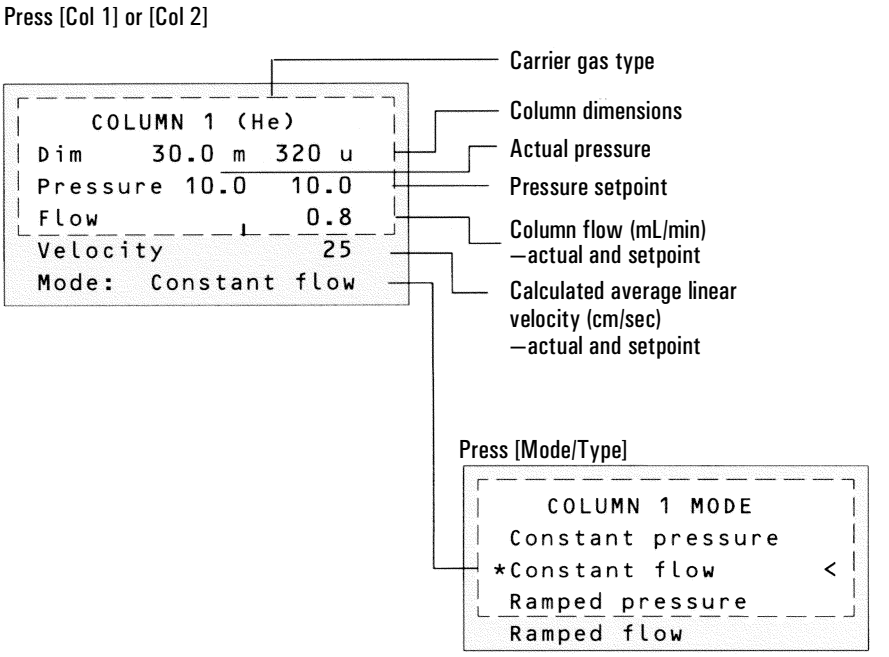
`Pressure` The setpoint appears at the far right. The number at the left shows the actual pressure value. When you enter a pressure value, the values for flow and average linear velocity are calculated and displayed.

`Flow` If you enter a flow (in mL/min) here, pressure and velocity are calculated and adjusted.

`Velocity` If you enter average linear velocity (in cm/sec), pressure and flow are calculated.

`Mode`: There are four column modes: constant flow, constant pressure, ramped flow, and ramped pressure. To change the mode, scroll to `Mode`: and press [Mode/Type].

"Flow and Pressure Control" explains how to set pressure and flow programs.



Mode: Your control table also has one of these, depending on Mode:

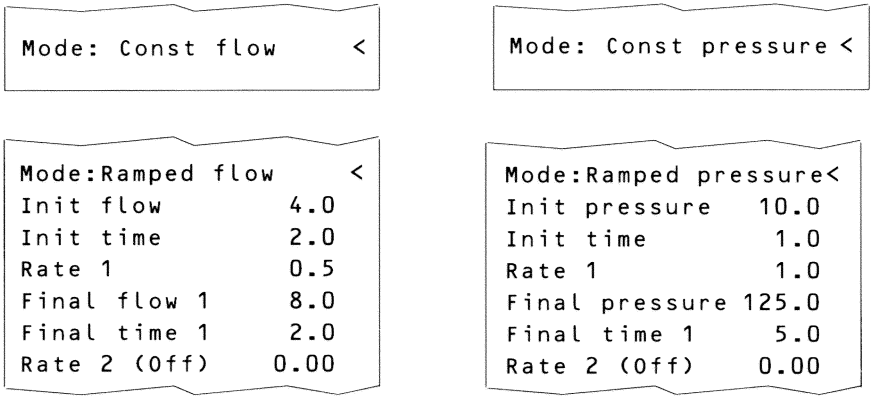


Figure 38 Column display — defined capillary columns

The column control table—packed or undefined capillary columns

If you have not defined your column or if your inlet selection is `Unspecified`, your column control table will be similar to Figure 39.

The title This heading identifies the column—Column 1 or Column 2— and the type of carrier gas configured to the inlet (in parentheses).

`Dimensions unknown` This line tells you that you have not defined your column.

`Pressure` The *split/splitless* inlet and the *cool on-column* inlet are pressure controlled. Because the column is unknown, flow and average linear velocity cannot be computed.

The *purged packed* inlet is flow controlled. The actual pressure is displayed, but is not controllable by the user.

`Mode:` You have a choice of three modes if using a split/splitless or cool on-column inlet—constant pressure, constant flow, and ramped flow. The packed inlet gives you only the two flow modes—constant and ramped.

"Flow and Pressure Control" explains how to set pressure and flow programs.

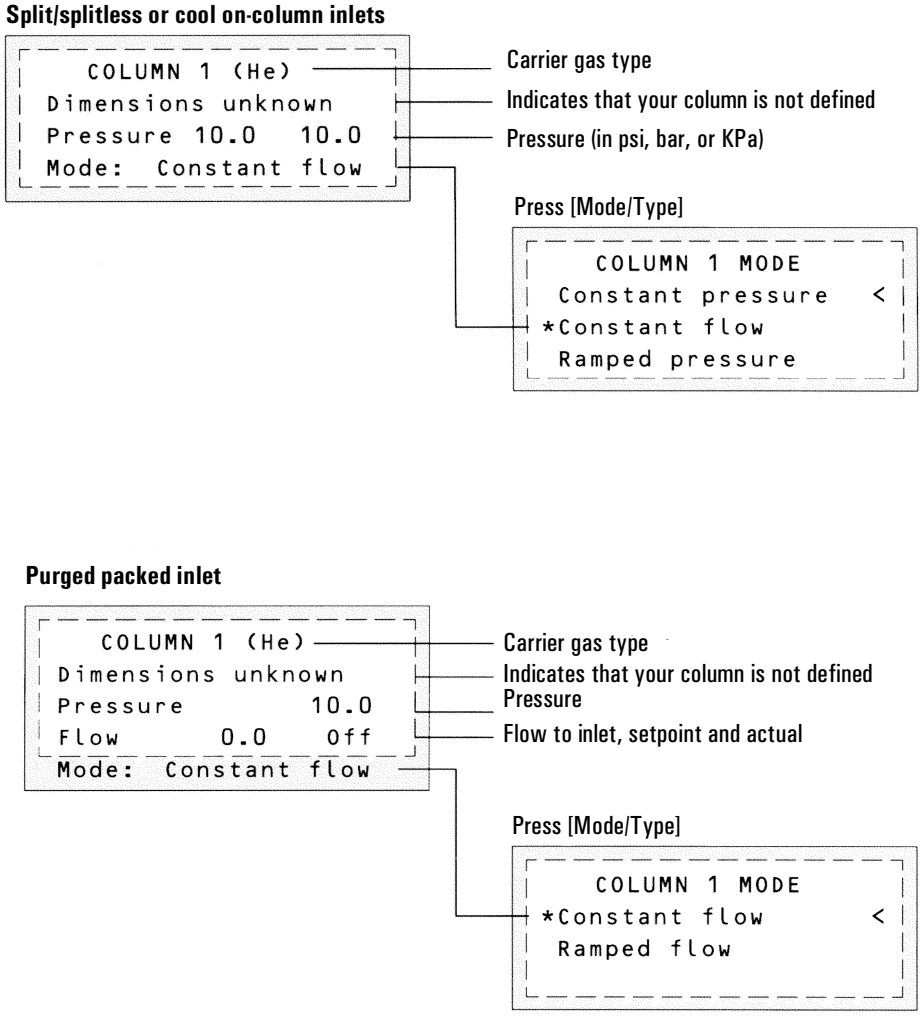


Figure 39 Column display — Packed or undefined capillary columns

What is gas saver?

Gas saver reduces carrier gas flow from the split vent after the sample is on the column. Column head pressure and flow rate are maintained, while purge and split vent flows decrease. Flows—except column flow—remain at the reduced level until you press [Prep Run].

You can use gas saver in all modes of operation of the Split/Splitless and PTV inlets and in the split and splitless modes of the Volatiles Interface.

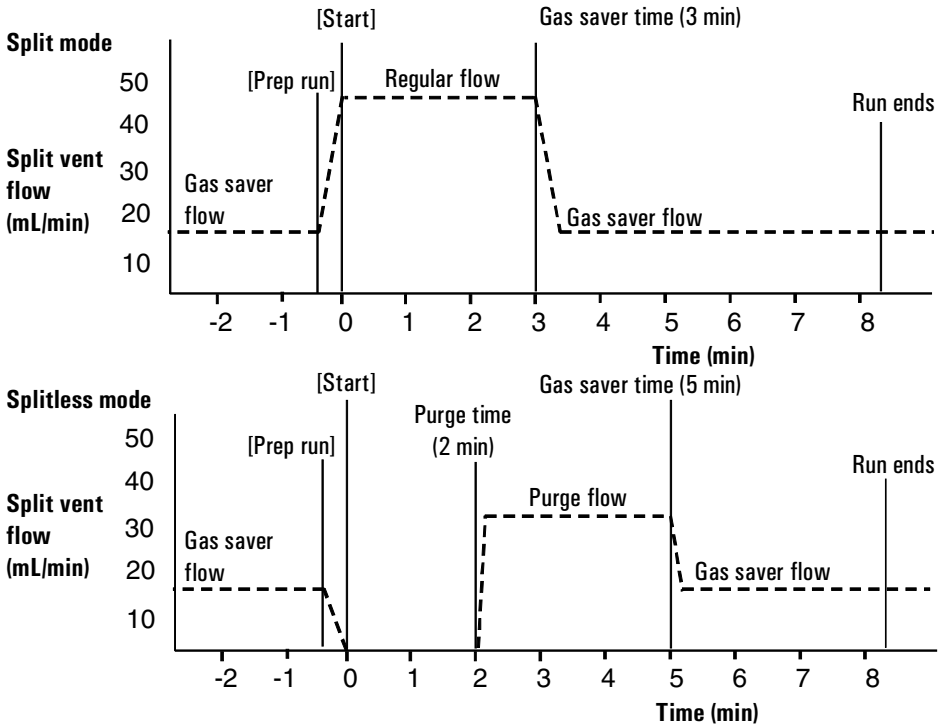
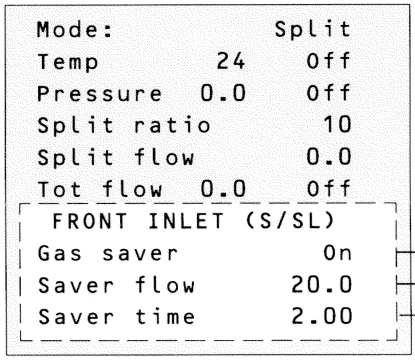


Figure 40 Gas saver operation

The pulsed modes of the split/splitless and PTV inlets are similar except for the pressure pulse starting at [Prep Run] and ending at `Pulse time`. The solvent vent mode of the PTV is more complex; see "Using the Solvent Vent Mode" for details.

Procedure: Using gas saver

Press [Front Inlet] or [Back Inlet].



1. Turn on gas saver.

2. Set a flow. Must be at least 15 mL/min greater than the column flow.

3. If in split mode, set after injection time. In all other modes, set after purge time.

Pre Run and Prep Run

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

You must use the Pre Run state when:

- Using gas saver with any inlet.
- Using splitless mode with any inlet.
- Using a pressure pulse mode with any inlet.
- Using the solvent vent mode of the PTV inlet.
- Using the direct or splitless mode of the Volatiles Interface.

There are two ways to begin Pre Run—manually push the [Prep Run] key before each run or configure the GC to enter the Pre Run state automatically. The two methods are discussed below.

During the Pre Run state:

- The Pre Run light blinks and Not Ready is on.
- Setpoints change to the correct values for injection.
- Inlet, detector, and oven equilibration times begin.

When all equilibration times expire, the Pre Run light is on steadily. When all criteria for a run are met, the Not Ready light turns off. The GC is now ready for sample injection.

The [Prep Run] key

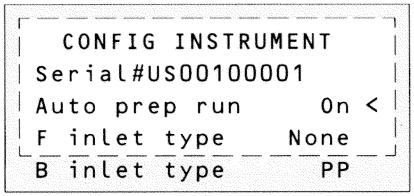
Press the [Prep Run] key before you inject a sample manually. The GC enters the Pre Run state. When the Pre Run light is steady and the Not Ready light goes off, begin the analysis.

Procedure: Auto Prep 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 GC to Auto Prep Run. To do this:

1. Press the [Config] key to view a list of configurable parameters.

2. Scroll to the `Instrument` parameter and press [Enter].
3. Scroll to `Auto prep run` and press [On].



Septum purge

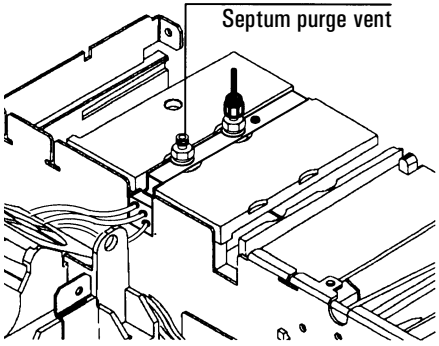
The septum purge line is near the septum where the sample is injected. A small amount of carrier gas exits through this line to sweep out any bleed.

Each inlet has a different septum purge flow. The GC automatically sets the purge flow for EPC inlets, but you can measure it from the septum purge vent at the flow manifold if you like.

Table 32 Septum Purge Flows

Inlet	Carrier	Septum purge (mL/min)
Split/splitless, all modes	He, N_2, Ar/5%Me	3
	H_2	6
Purged packed	All	1 to 3
Cool on-column	He, N_2, Ar/5%Me	15
	H_2	30
PTV	He, N_2, Ar/5% Me	3
	H_2	6
Volatiles interface	He, N2, Ar/5%Me	3
	H_2	6

Split/splitless inlet
PTV inlet
Violates interface



Purged packed inlet
Cool on-column inlet

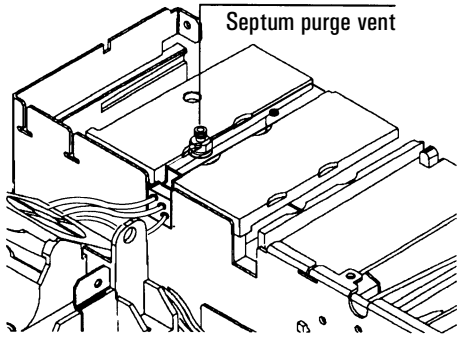


Figure 41 Septum purge vents

14 The Split/Splitless Inlet

Using a Split/Splitless Inlet

Standard and high-pressure versions
Septum tightening

Liners
Procedure: Changing the liner

Columns and Traps

Split mode pneumatics

The control table—split operation
Procedure: Using the split mode with the column defined
Procedure: Using the split mode with the column not defined

Splitless mode pneumatics

The control table—splitless operation

Operating parameters
Procedure: Using splitless mode with the column defined
Procedure: Using splitless mode with the column not defined

Pulsed split and splitless modes

The control table—pulsed split mode
Procedure: Using the pulsed split mode

The control table—pulsed splitless operation
Procedure: Using the pulsed splitless mode

Maintaining a split/splitless inlet

Changing septa
Procedure: Changing the septum

Changing the O-ring
Procedure: Changing the O-ring

Replacing the inlet base seal
Procedure: Replacing the inlet base seal
Replacing the split vent trap filter cartridge
Procedure: Leak testing the gas plumbing
Procedure: Leak testing an EPC split/splitless inlet
Procedure: Leak testing a nonEPC split/splitless inlet
Procedure: Correcting leaks
Procedure: Cleaning the inlet

The Split/Splitless Inlet

Using a Split/Splitless Inlet

This inlet is used for split, splitless, pulsed splitless, or pulsed split analyses. You can choose the operating mode from the inlet control table. The *split mode* is generally used for major component analyses, while the *splitless mode* is used for trace analyses. The *pulsed splitless* and *pulsed split modes* are used for the same type of analyses as split or splitless, but allow you to inject larger samples.

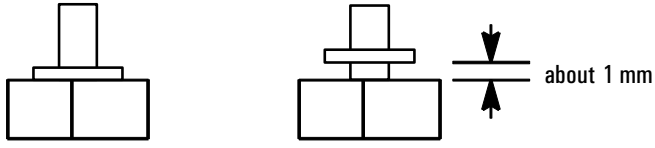
Standard and high-pressure versions

The standard split/splitless inlet is rated to 120 psi pressure at the gas supply fitting. It is appropriate for most columns. The high-pressure inlet is rated to 170 psi pressure—it is useful with very small diameter capillary columns that offer considerable resistance to gas flow.

To determine the version that you have, press [Front Inlet] or [Back Inlet], scroll to the `Pressure` line, and press the [Info] key. The display will show the pressure range for the inlet—either `1 to 100 psi` (for the standard version) or `1 to 150 psi` (for the high-pressure version).

Septum tightening

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



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

If using a Merlin Microseal™ septum, finger tighten the septum nut, until snug (not loose). The pressure capacity depends on the septum used.

Liners

Choose liners according to the type of injection you are doing—split or splitless. Many liners are available and can be ordered from the Agilent catalog for consumables and supplies.

Procedure: Changing the liner

Parts list:

- Liner, part no. 5183-4647 (split) or 5062-3587 (splitless)
- Tweezers
- Septum wrench (part no. 19251-00100)
- Viton O-ring (part no. 5180-4182)

1. Press [Oven] and set the oven to 35°C. When the temperature reaches setpoint, turn the oven off. Press [Front Inlet] or [Back Inlet] and turn off the inlet temperature and pressure.

WARNING Be careful! The inlet fittings may be hot enough to cause burns.

2. Remove the insert retainer nut. Use a septum wrench, if needed.
3. If a liner is present, remove it with tweezers or a similar tool. Be careful not to chip the liner.
4. Hold the new liner with tweezers, and inspect it. Make sure it is the correct type for the injection mode you are using—split or splitless.
5. Place a Viton O-ring on the liner about 2 to 3 mm from its top end.
6. Press the liner straight down into the inlet.

Caution Do not add an O-ring or other seal either at the bottom of the inlet or at the bottom of the liner; this will damage the inlet and shatter the liner.

7. Replace the insert retainer nut, tightening it to firm finger tightness. Do not overtighten.

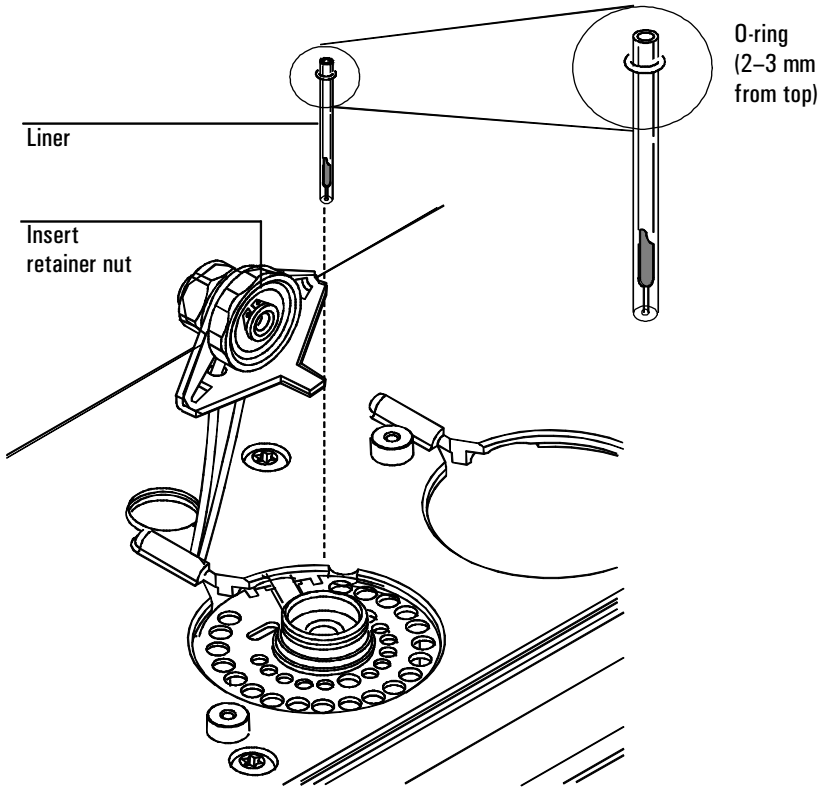


Figure 42 Installing a liner

Split mode pneumatics

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

Figure 43 shows the pneumatics for this inlet in split mode operation.

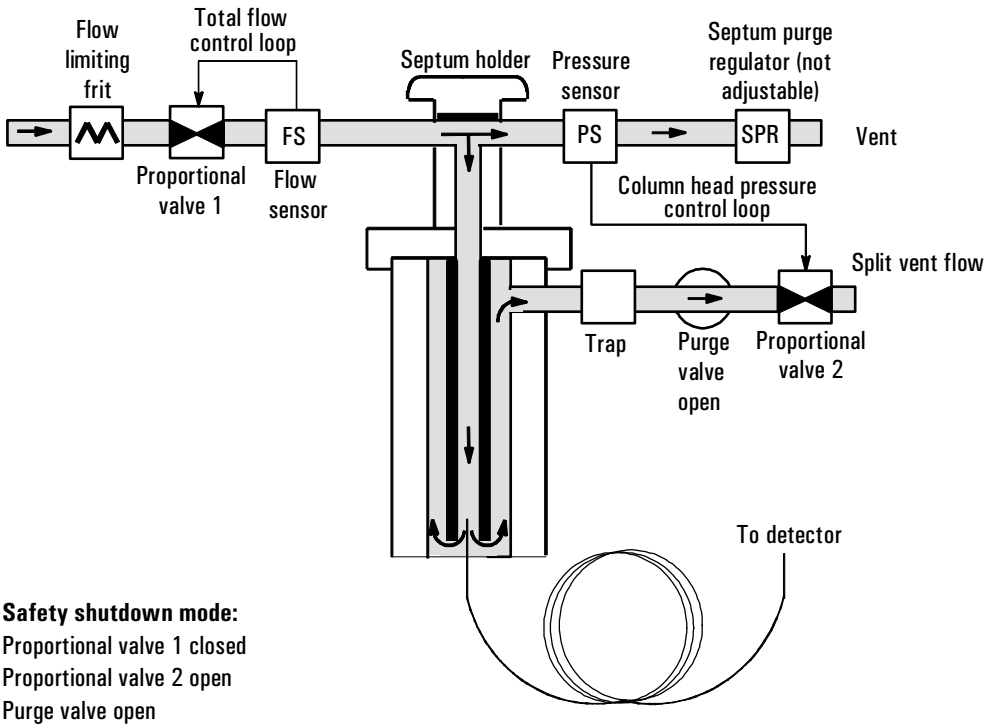


Figure 43 Split flow pneumatics

The control table—split operation

`Mode:` The current operating mode—split

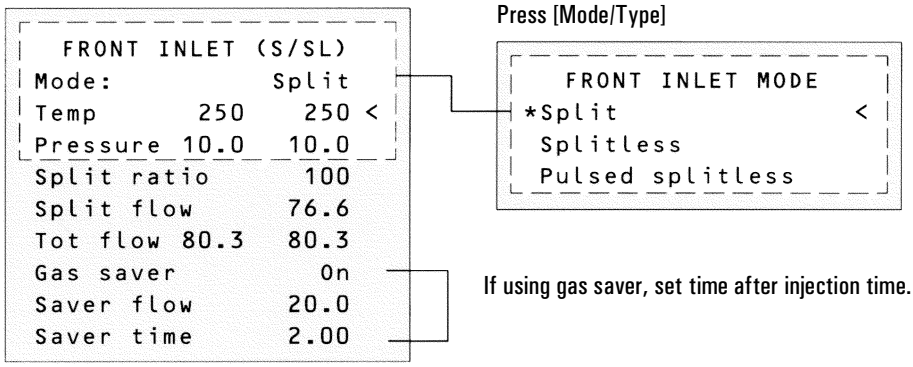
`Temp` Actual and setpoint inlet temperatures

`Pressure` Actual and setpoint inlet pressure

`Split ratio` The ratio of split flow to column flow. Column flow is set at the Column 1 or Column 2 control table. This line does not appear if your column is not defined.

`Split flow` Flow, in mL/min, from the split/purge vent. This line does not appear if your column is not defined.

`Total flow` This is the total flow into the inlet, which is the sum of the split flow, column flow, and septum purge flow. When you change the total flow, the split ratio and split flow change while the column flow and pressure remain the same.



Procedure: Using the split mode with the column defined

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".

2. Press [Front Inlet] or [Back Inlet]

   a. Scroll to `Mode:` and press [Mode/Type]. Select `Split`.

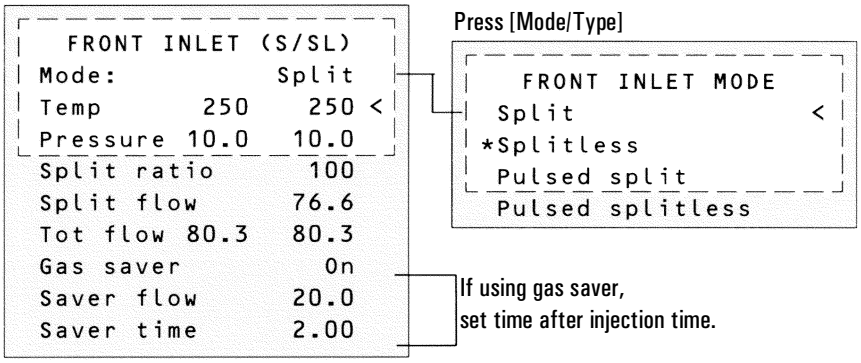
   b. Set the inlet temperature.

   c. If you want a specific split ratio, scroll to `Split ratio` and enter that number. The split flow will be calculated for you.

   d. If you want a specific split flow, scroll to `Split flow` and enter that number. The split ratio will be calculated for you.

   e. If desired, turn on `Gas saver`. Set the `Saver time` after the injection time. Use the [Prep Run] key (see page 285) before manually injecting the sample.

$$\text{Split ratio} = \frac{\text{Split flow}}{\text{Column flow}}$$



Procedure: Using the split mode with the column not defined

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".

2. Press [Front Inlet] or [Back Inlet]

```
  FRONT INLET (S/SL)
Mode:            Split
Temp       250    250 <
Pressure  10.0   10.0
Tot flow  79.1   79.1
```

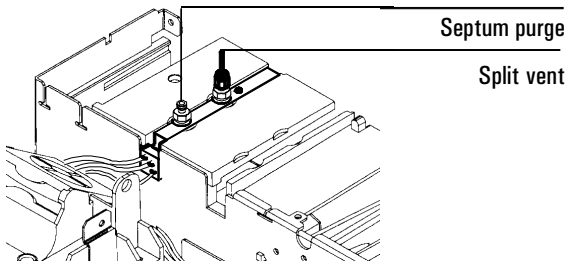
   a. Set temperature.

   b. Set total flow into the inlet. Measure flow out of the split vent using a flow meter.

   c. Subtract split vent flow and septum purge flow (see page 286 for nominal septum purge flows by carrier gas type) from `Total flow` to get column flow.

   d. Calculate the split ratio. Adjust as needed.

$$\text{Split ratio} = \frac{\text{Split flow}}{\text{Column flow}}$$



Front of instrument

Splitless mode pneumatics

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

If you are using gas saver, the gas saver time should be *after* the purge time.

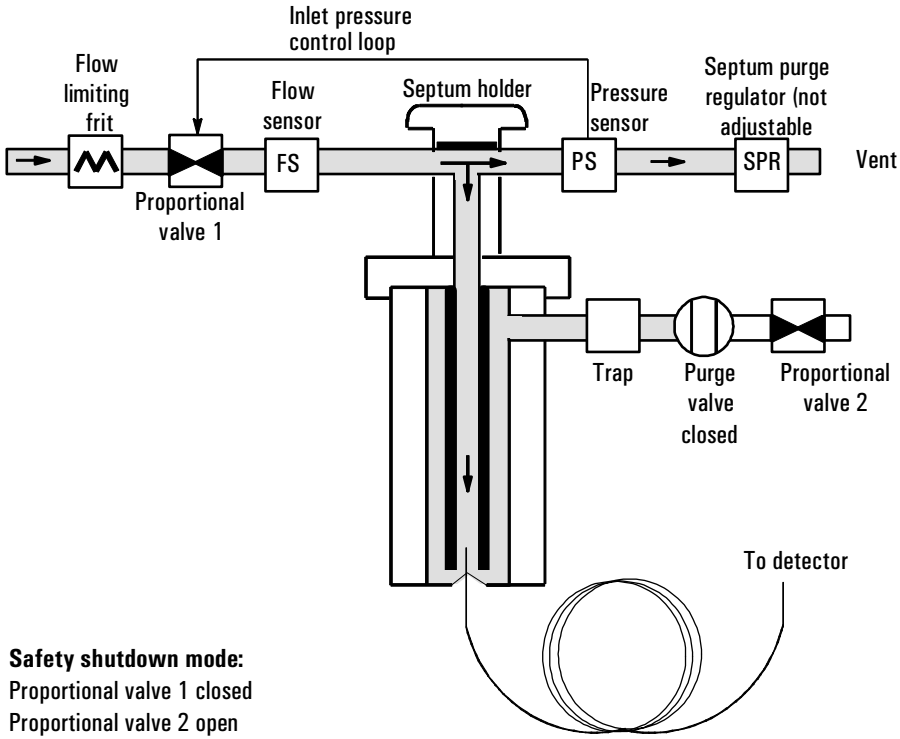


Figure 44 Splitless flow diagram, pre-run to purge time

The control table—splitless operation

`Mode:` The current operating mode—splitless

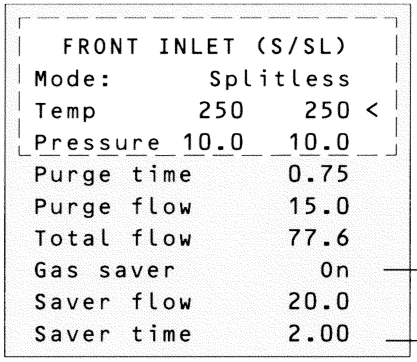
`Temp` Actual and setpoint inlet temperatures

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

`Purge time` The time, after the beginning of the run, when you want the purge valve to open.

`Purge flow` The flow, in mL/min, from the purge vent, at `Purge time`. You will not be able to specify this value if operating with your *column not defined*.

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



Operating parameters

A successful splitless injection consists of these steps:

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

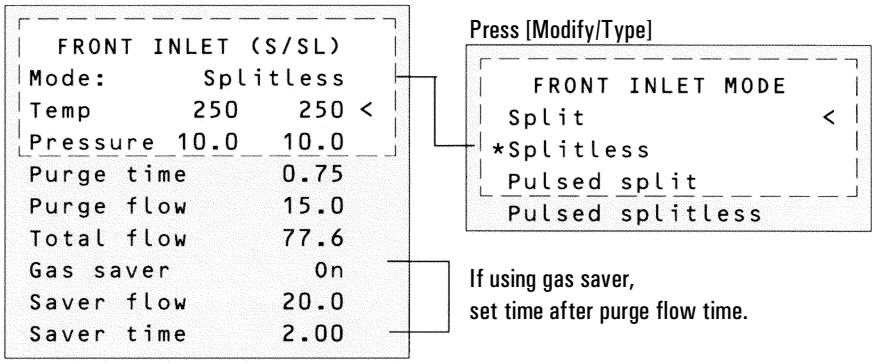
Some experimentation is needed to refine the operating conditions. Table 33 provides starting values for the critical parameters.

Table 33 Splitless Mode Inlet Parameters

Parameter	Allowed setpoint range	Suggested starting value
Oven temperature	No cryo, 24° C to 450° C CO_2 cryo, –60° C to 450° C N_2 cryo, –80° C to 450° C	10°C below solvent boiling point
Oven initial time	0 to 999.9 minutes	≥ Inlet purge time
Inlet purge time	0 to 999.9 minutes	$\frac{\text{Liner volume}}{\text{Column flow}} \times 1$
Gas saver time	0 to 999.9 minutes	After purge time
Gas saver flow	15 to 1000 mL/min	15 mL/min greater than maximum column flow

Procedure: Using splitless mode with the column defined

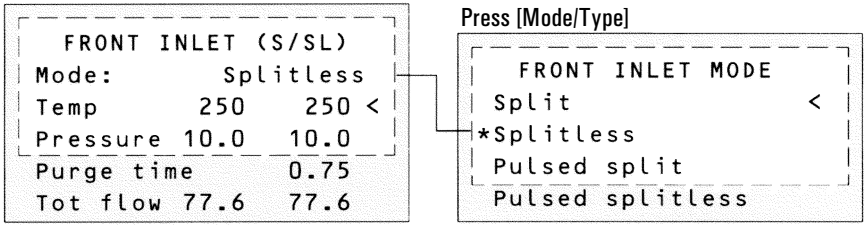
1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet] or [Back Inlet]
   a. Scroll to `Mode`: and press [Mode/Type]. Select `Splitless`.
   b. Set the inlet temperature.
   c. Enter a purge time and a purge flow.
   d. If desired, turn `Gas saver` on. Make certain the time is set *after* the purge flow time.



3. Use the [Prep Run] key (see page 285) before manually injecting a sample.

Procedure: Using splitless mode with the column not defined

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet] or [Back Inlet]
   a. Scroll to `Mode`: and press [Mode/Type]. Select `Splitless`.
   b. Set the inlet temperature.
   c. Enter a purge time.
   d. Set your total flow greater than the column flow plus the septum purge flow—see page 286—to guarantee adequate column flow.



3. Use the [Prep Run] key (see page 285) before manually injecting a sample.

Pulsed split and splitless modes

The pressure pulse modes increase inlet pressure just before the beginning of a run and returns it to the normal value after a specified amount of time. The pressure pulse sweeps the sample out of the inlet and into the column faster, reducing the chance for sample decomposition in the inlet. If your chromatography is degraded by the pressure pulse, a retention gap may help restore peak shape.

You must press the [Prep Run] key before doing manual injections in the pressure pulse mode. See page 285 for details.

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

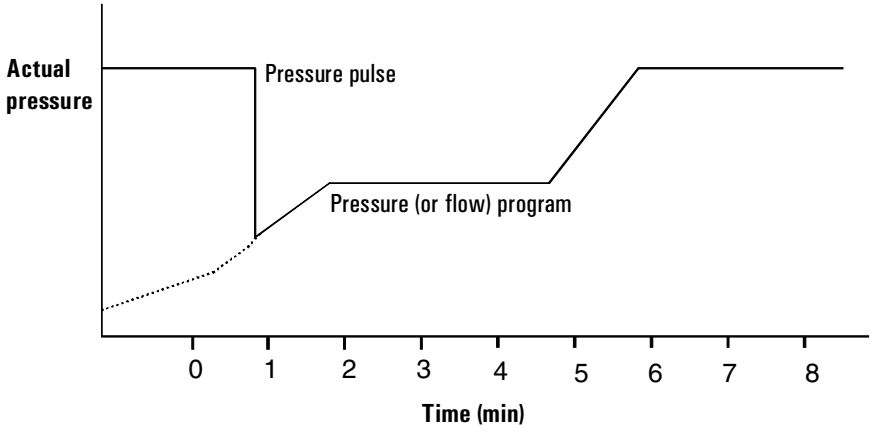


Figure 45 Pressure pulse and column flow or pressure

The control table—pulsed split mode

`Mode:` The current operating mode—pulsed split

`Temp` Actual and setpoint inlet temperatures

`Pressure` Actual and setpoint inlet pressure at the beginning of a run, ignoring the effect of a pressure pulse. It sets the starting point of a pressure program or the fixed pressure if a program is not used.

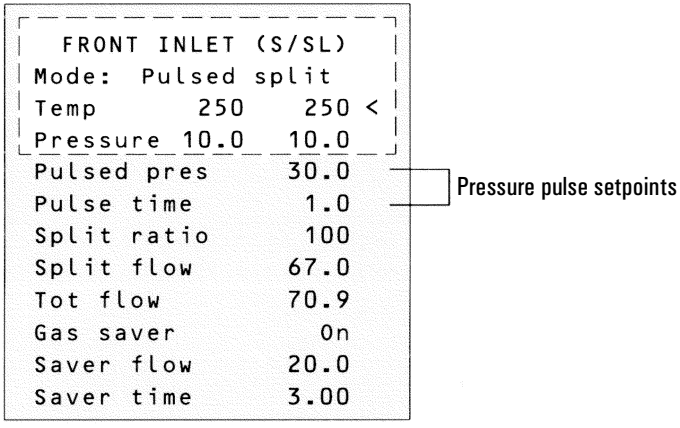
`Pulsed pres` The inlet pressure you desire at the beginning of a run. The pressure rises to this setpoint after [Prep Run] is pressed and remains constant until `Pulse time` elapses, when it returns to `Pressure`.

`Pulse time` Pressure returns to its normal setpoint at this time.

`Split ratio` The ratio of split flow to column flow. Column flow is set at the Column 1 or 2 control table. Appears only if the column is defined.

`Split flow` Flow, in mL/min from the split/purge vent. Appears only if the column is defined.

`Total flow` The sum of the split flow, column flow, and septum purge flow. If you change the total flow, the split ratio and split flow change while the column flow and pressure remain the same. When a pressure pulse is used, total flow increases to keep the split ratio constant.

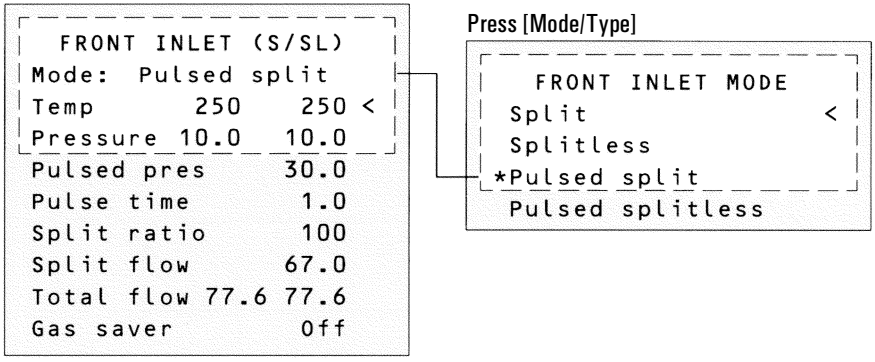


Procedure: Using the pulsed split mode

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet] or [Back Inlet]
   a. Scroll to `Mode:` and press [Mode/Type]. Select `Pulsed Split`.
   b. Set the inlet temperature.
   c. Enter values for `Pulsed Pres` and `Pulse time`.

$$\text{Split ratio} = \frac{\text{Split flow}}{\text{Column flow}}$$

   d. If you want a specific split ratio, scroll to `Split ratio` and enter that number. The split flow is calculated for you if the column is defined.
   e. If you want a specific Split flow, scroll to `Split flow` and enter that number. The split ratio is calculated for you if the column is defined.
   f. Turn `Gas saver` on, if desired. Make certain the time is set *after* `Pulse time`.



3. Press the [Prep Run] key (see page 285) before injecting a sample manually.

The control table—pulsed splitless operation

`Mode:` The current operating mode—pulsed splitless

`Temp` Actual and setpoint inlet temperatures

`Pressure` Actual and setpoint inlet pressure at the beginning of a run, ignoring the effect of a pressure pulse. It sets the starting point of a pressure program or the fixed pressure if a program is not used.

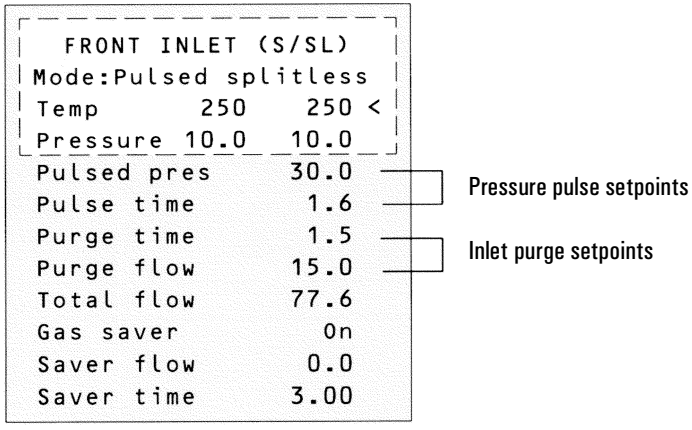
`Pulsed pres` The inlet pressure you desire at the beginning of a run. The pressure rises to this setpoint after [Prep Run] is pressed and remains constant until `Pulse time` elapses, when it returns to `Pressure`.

`Pulse time` Pressure returns to its normal setpoint at this time.

`Purge time` The time, after the beginning of the run, that you wish the purge valve to open. Set purge time 0.1 to 0.5 minutes before pulse time.

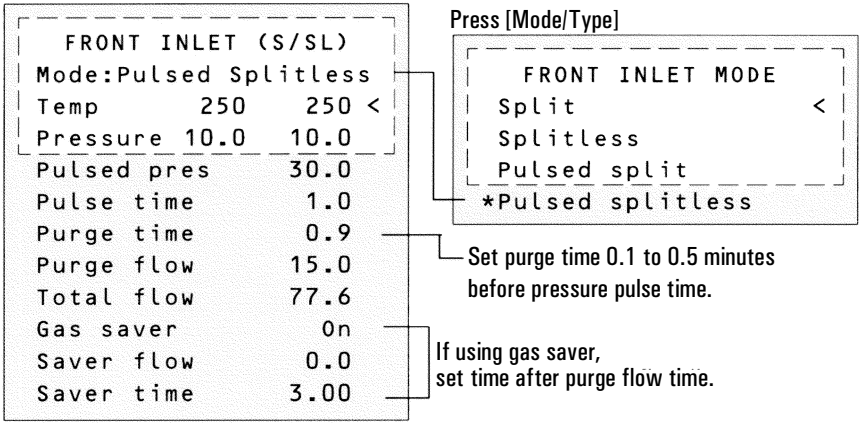
`Purge flow` The flow, in mL/min, from the purge vent, at `Purge time`. The column must be defined.

`Total flow` This is the total flow into the inlet, representing a total of the column flow and the septum purge flow.



Procedure: Using the pulsed splitless mode

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet] or [Back Inlet]
   a. Scroll to `Mode`: and press [Mode/Type]. Select `Pulsed Splitless`.
   b. Set the inlet temperature.
   c. Enter values for `Pulsed pres` and `Pulse time`.
   d. Enter the Purge time when you wish the purge valve to open. Set 0.1 to 0.5 minutes before `Pulse time`.
   e. If your column is defined, enter a `Purge flow`.
   f. If your column is defined, turn `Gas saver` on, if desired. Make certain the time is set *after* the purge flow time.



3. Press the [Prep Run] key (see page 285) before injecting a sample manually.

Maintaining a split/splitless inlet

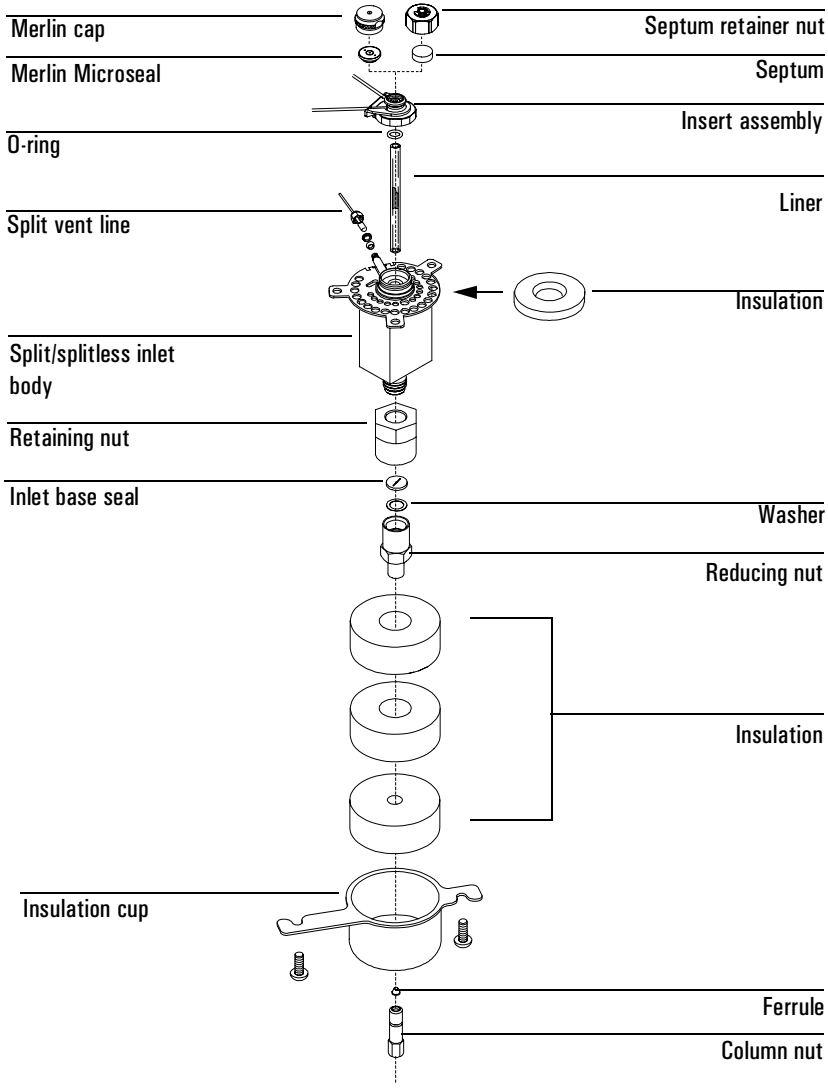


Figure 46 The split/splitless capillary inlet

Changing septa

If a septum leaks, you will see symptoms such as longer or shifting retention times, loss of response, and/or loss of column head pressure. Additionally, signal noise will increase.

The useful lifetime of septa depends upon injection frequency and needle quality; burrs, sharp edges, rough surfaces, or a blunt end on the needle decrease septum lifetime. When the instrument is in steady use, daily septum replacement is recommended.

The type of septa you use will depend on your chromatography needs. Another available option is the Merlin Microseal™ septum, a duckbill septum providing low bleed and longer life when used with the 7683 automatic Sampler and recommended syringes. You can order septa directly from Agilent Technologies; refer to the Agilent catalog for consumables and supplies for ordering information.

Table 34 Recommended Septa for the Split/Splitless Inlet

Description	Part no.
11-mm septum, low-bleed red	5181-1263
11-mm septum with partial through-hole, low-bleed red	5181-3383
11-mm septum, low-bleed gray	5080-8896
Merlin Microseal septum (30 psi)	5181-8815
11-mm high-temperature silicon septum (350° C and higher)	5182-0739

WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

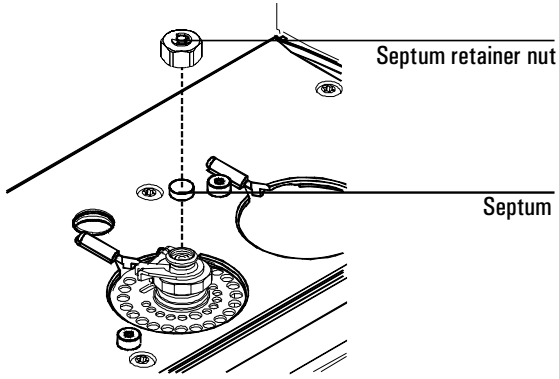
Procedure: Changing the septum

Materials needed:

- Gloves (if inlet is hot)
- New septum—see Table 34 on page 307 for part numbers
- Septum nut wrench (part no. 19251-00100)
- A plastic or wood tool with a sharp tip to remove septum from inlet
- 0- or 00-grade steel wool (optional)
- Forceps or tweezers
- Compressed, filtered, dry air or nitrogen (optional)

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Turn the oven and detector off.
   - Cool the oven and inlet to room temperature.
   - Turn the inlet pressure off.
2. Remove the septum retainer nut or Merlin cap, using the wrench if the nut is hot or sticks. Remove the old septum or Merlin Microseal. If the septum sticks, use a sharp tool to remove it. Be sure to get all of it. Take care to avoid gouging or scratching the interior of the septum head.

If the septum sticks, use the sharp-tipped tool to remove it. Take care not to gouge the metal around the septum, and remove all pieces of the old septum

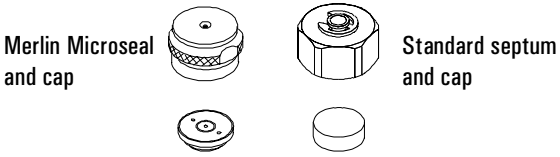


3. If pieces of the septum are sticking, use a small piece of rolled-up steel wool and forceps or tweezers to scrub the residue from the retainer nut and septum

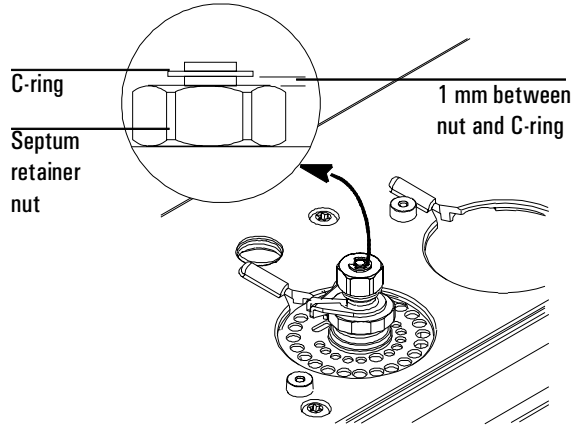
holder. Use compressed air or nitrogen to blow away the pieces of steel wool and septum.

4. Use forceps to insert a new septum or Merlin Microseal. Press it into the fitting firmly.

   If installing a Merlin Microseal, install it so that the side with the metal parts faces down (toward the oven).



5. Replace the septum retainer nut or Merlin cap, tightening it finger-tight. If using the standard septum retainer nut, the C-ring is about 1 mm above the nut. Avoid overtightening.



6. Restore normal operating conditions.

Changing the O-ring

You will need to change the O-ring each time you change the liner, or if it wears out and becomes a source of leaks in the inlet. To determine if the O-ring leaks, run the leak test for the split/splitless inlet.

O-rings contain plasticizers that give them elasticity. The O-ring seals the top of the inlet, the inlet base, and the liner. However, at high temperatures the plasticizers bake out, and the O-rings become hard and are no longer able to create a seal.

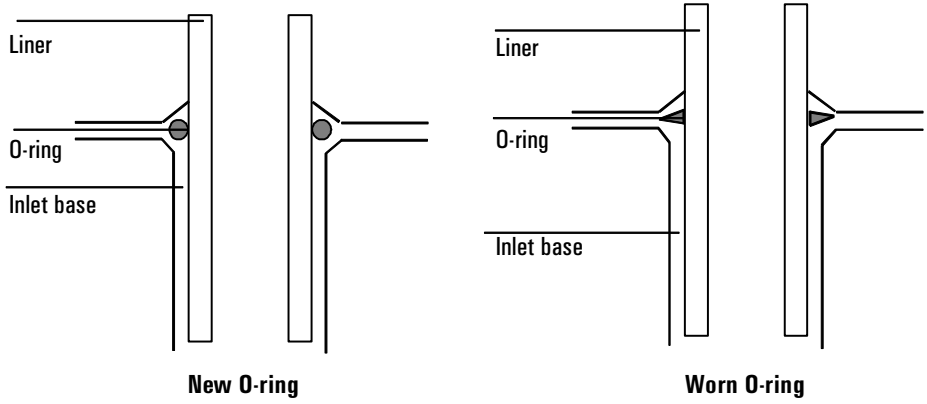


Figure 47 Cross section of inlet, liner, and O-ring

If you regularly operate the inlet at high temperatures, you may want to use graphite O-rings. Although they have a longer life-time, they too will eventually take a set. Refer to the table below to make sure you are using the correct O-ring for your inlet.

Table 35. O-Rings for the Split/Splitless Inlet

Description	Part no.
Viton O-ring for temperatures up to 350° C	5181-4182
Graphite O-ring for split liner (temperatures above 350° C)	5180-4168
Graphite O-ring for splitless liner (temperatures above 350° C)	5180-4173

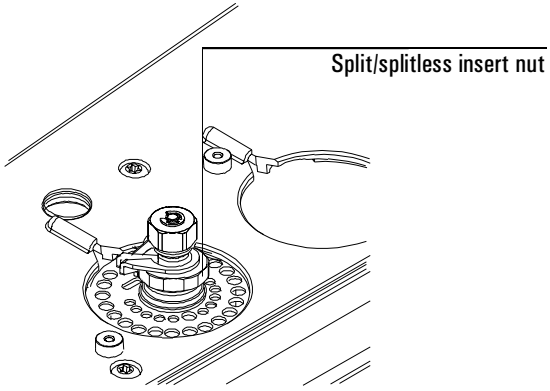
WARNING Be careful! The oven and/or inlet may be hot enough to cause burns. If the inlet is hot, wear gloves to protect your hands.

Procedure: Changing the O-ring

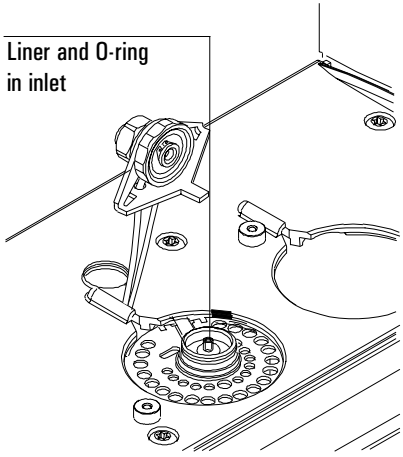
Materials needed:

- Gloves (if inlet is hot)
- A new O-ring—refer to Table 35 on page 310
- Septum nut wrench (part no. 19251-00100)
- Forceps or tweezers

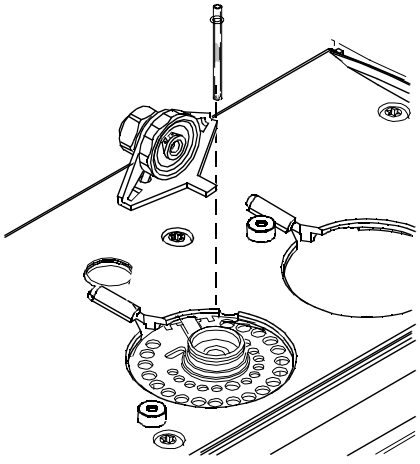
1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Turn the oven and detector off.
   - Cool the oven and inlet to room temperature.
   - Turn the inlet pressure off.
2. Locate the split/splitless insert nut and loosen it using the wrench if necessary. Lift it straight up to avoid chipping or breaking the liner.



3. You should see the top of the liner with the O-ring around it. Use the forceps or tweezers to grasp the liner and pull it out.



4. Remove the old O-ring and slide a new one onto the liner.
5. Use the forceps to return the liner to the inlet. Replace the insert assembly nut and use the wrench to tighten the nut just to snugness.



6. Restore the GC to normal operating conditions.

Replacing the inlet base seal

You must replace the inlet base seal whenever you loosen or remove the reducing nut. In addition, chromatographic symptoms such as ghost peaks indicate that the inlet base seal is dirty and should be replaced.

Three types of inlet base seals are available:

- Gold-plated seal, part no. 18740-20885
- Gold-plated seal, cross, part no. 5182-9652
- Stainless steel seal, part no. 18740-20880

You change the inlet base seal from inside the oven, so you must remove the column. If you are unfamiliar with column installation and removal, see "Columns and Traps".

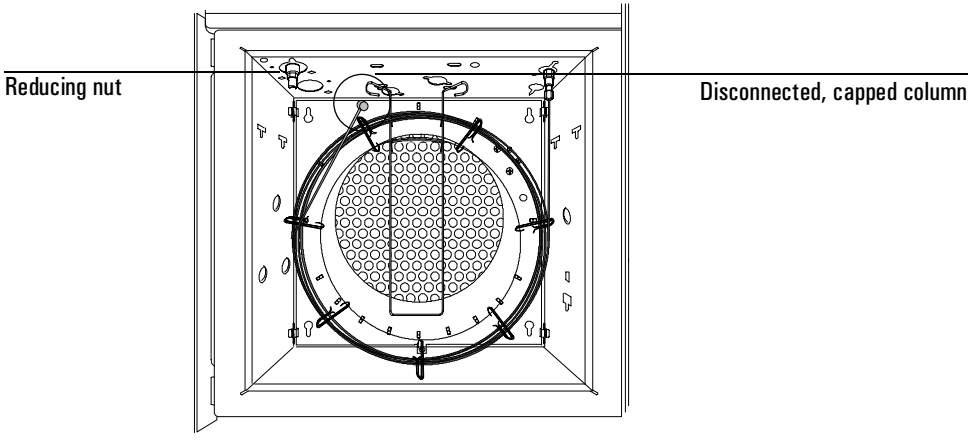
WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

Procedure: Replacing the inlet base seal

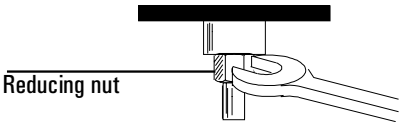
Materials needed:

- Clean, lint-free, non-nylon gloves (must wear when handling seal)
- A new seal (see list of part numbers)
- A new washer (part no. 5061-5869)
- 1/4-inch wrench (for column)
- 1/2-inch wrench

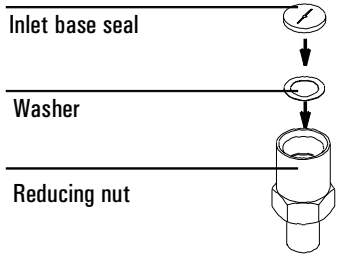
1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Turn the oven and detector off.
   - Cool the oven and inlet to room temperature.
   - Turn the inlet pressure off.
2. Remove the column from the inlet. Cap the open end of the column to prevent contamination. If an insulation cup is installed around the base of the inlet, remove it.



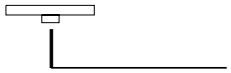
3. Use the 1/2-inch wrench to loosen the reducing nut, and then remove it. The washer and seal are inside the reducing nut. Remove them. You will probably want to replace the washer when you replace the inlet seal.



4. Put on the gloves to protect the inlet base seal and washer from contamination. Place the washer in the reducing nut. Place the new inlet base seal on top of it.



Side view of inlet base seal:



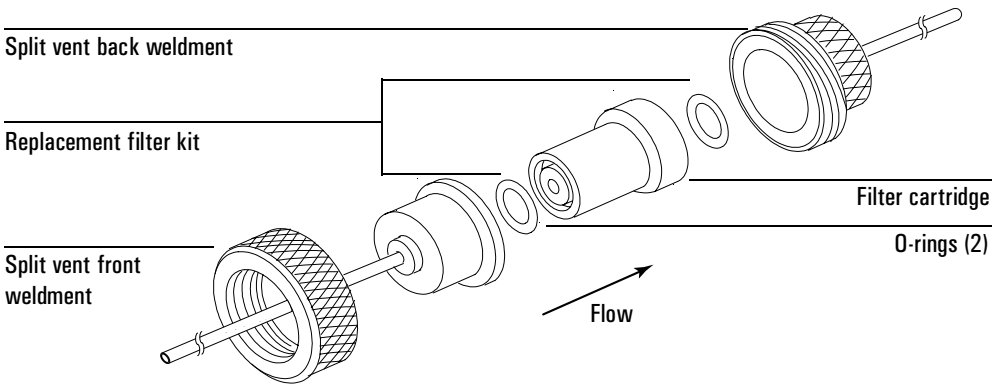
5. Replace the reducing nut. Use the 1/2-inch wrench to tighten the nut. Replace the column and the insulation cup. After the column is installed, you can restore normal operating conditions.

Replacing the split vent trap filter cartridge

WARNING Turn off the oven and turn off the heater for the inlet that uses the split vent trap and let them cool down. Turn off the carrier gas supply pressure.

The split vent trap may contain residual amounts of any samples or other chemicals you have run through the GC. Follow appropriate safety procedures for handling these types of substances while replacing the trap filter cartridge.

1. Turn off the inlet and the oven and allow to cool.
2. Set all GC flows to zero.
3. Remove the pneumatics cover.
4. Lift the filter trap assembly form the mounting bracket and unscrew the filter trap assembly.



5. Remove the old filter cartridge and O-rings and replace them.
6. Reassemble the trap.
7. Check for leaks.

Procedure: Leak testing the gas plumbing

Leaks in the gas plumbing can affect chromatographic results dramatically. The following procedure checks the flow system up to but not including the inlet flow manifold. If this portion of the system proves to be leak-free, refer to the next procedure to check the inlet and inlet manifold.

Liquid leak detectors are not recommended, especially in areas where cleanliness is very important.

If you do use leak detection fluid, immediately rinse the fluid off to remove the soapy film.

WARNING To avoid a potential shock hazard when using liquid detection fluid, turn the GC off and disconnect the main power cord. Be careful not to spill leak solution on electrical leads, especially the detector heater leads.

Materials needed:

- Electronic leak detector capable of detecting your gas type or liquid leak detection fluid. If you use leak detection fluid, remove excess fluid when you have completed the test.
- Two 7/16-inch wrenches

1. Using the leak detector, check each connection you have made for leaks.
2. Correct leaks by tightening the connections. Retest the connections; continue tightening until all connections are leak-free.

Procedure: Leak testing an EPC split/splitless inlet

There are numerous places in the inlet that can leak. This procedure lets you determine, in general, if there is an unacceptable leak in the inlet.
If the inlet is leaking, you should use an electronic leak detector to pinpoint the component that is leaking.

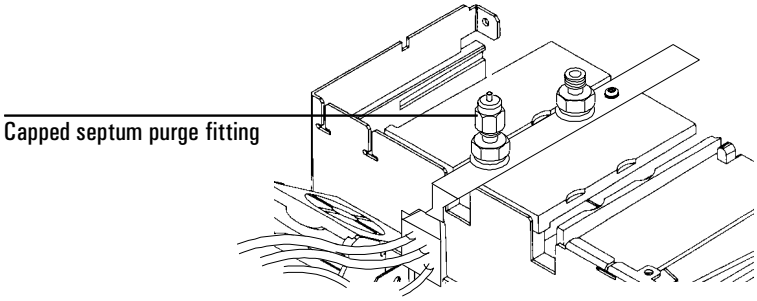
WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

Materials needed:

- No-hole ferrule
- 7/16-inch wrench
- Gloves (if the inlet is hot)
- Septum nut wrench (part no. 19251-00100)
- 9/16-inch wrench
- 1/8-inch SWAGELOK cap
- Bubble flow meter

1. Complete the following preliminary steps:
    - If you have entered parameters that you do not want to lose, store them as a method.
    - Turn the oven off.
    - Cool the oven and inlet to room temperature.
    - Turn the inlet pressure off.
    - Remove the column, if one is installed, and plug the column fitting with the column nut and a no-hole ferrule.
    - Remove the old septum and replace it with a new one. For instructions, see "Changing septa".
    - Inspect the O-ring and replace it if it is hard and brittle or cracked. See "Changing the O-ring" for instructions.

2. Cap the septum purge fitting with a 1/8-inch SWAGELOK cap.



3. Set the oven to its normal operating temperature.
4. Configure the column as 0 length.
5. Press [Front Inlet] or [Back Inlet] to open the inlet's control table.
   - Set the inlet to its normal operating temperature.
   - Enter a pressure setpoint of 25 psi, or enter your normal operating pressure if it is greater. Make sure that the pressure at the gas supply is at least 10 psi higher than the inlet pressure.
   - Set the total flow to 60 mL/min.
   - Set the inlet to Split Mode.

   Wait a few moments for the pressure and flow to equilibrate. If pressure cannot be achieved, there is either a large leak or the supply pressure is to low.
6. Turn either the pressure or the flow off. Because the septum purge and the column fittings are capped, gas should be trapped in the system and the pressure should remain fairly constant.
7. Monitor the pressure for 10 minutes. A pressure drop of less than 0.5 psig (0.05 psi/min or less) is acceptable.

   If the pressure drops much faster than the acceptable rate, see "Procedure: Correcting leaks".

Procedure: Leak testing a nonEPC split/splitless inlet

There are numerous places in the inlet that can leak. This procedure lets you determine, in general, if there is an unacceptable leak in the inlet.
If the inlet is leaking, you should use an electronic leak detector to pinpoint the component that is leaking.

WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

Materials needed:

- No-hole ferrule
- 7/16-inch wrench
- Gloves (if the inlet is hot)
- Septum nut wrench (part no. 19251-00100)
- 9/16-inch wrench
- 1/8-inch SWAGELOK cap
- Bubble flow meter

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Cool the oven to room temperature and then turn it off.
   - When the oven is cool, turn off the inlet pressure.
   - Remove the column, if one is installed, and plug the column fitting with the column nut and a no-hole ferrule.
   - Remove the old septum and replace it with a new one. For instructions on changing septa, see page 307.
   - Inspect the O-ring and replace it if it is hard and brittle or cracked. See page 310 for instructions.
2. Cap the purge vent with a 1/8-inch SWAGELOK cap.
3. Set the oven to its normal operating temperature.

4. Set the inlet to its normal operating temperature. Make sure that the pressure at the initial gas supply is at least 35 psi.
5. Set the inlet pressure to 25 psi, or to your normal operating pressure, if it is higher. Set the split flow to 60 mL/min. Wait a few moments for the pressure and flow to equilibrate. If the system cannot reach the pressure setting, their either is a large leak or the supply pressure is too low.
6. Verify that the split flow is off by using a bubble flow meter.
7. Turn off flow to the inlet by turning off the carrier gas at the flow controller. Then, adjust the back pressure regulator clock-wise an additional 1/2 turn.

   Observe the column pressure for approximately 10 minutes. If the pressure drops less than 0.5 psig (0.5 psi/min or less), you can consider the inlet leak-free.

   If the pressure drops much faster than the acceptable rate, go to the next section, “Correcting Leaks.”

Procedure: Correcting leaks

Materials needed:

- Electronic leak detector
- Tools to tighten connections

1. Use the electronic leak detector to check all areas of the inlet that are potential sources of a leak. Potential leak areas are:
   - The capped purge vent
   - The plugged column connection
   - The septum and/or septum nut
   - The area where the gas lines are plumbed to the inlet—the O-ring, the O-ring nut, and the inlet base seal.
2. Correct leaks using the correct size wrench to tighten connections. You may need to repeat the leak test again to check for leaks.

   If the pressure drop is now 0.03 psi/min or less, you can consider the inlet system leak-free. If the pressure drops faster than this, continue to search for leaks and repeat the pressure test. If all fittings appear to be leak free, but the inlet system is still losing too much pressure, you may need to replace the inlet manifold. Contact your Agilent service representative.

Procedure: Cleaning the inlet

It is unlikely that the inlet will frequently require the thorough cleaning that this procedure presents; however, deposits from injected samples occasionally build up inside the split/splitless inlet. Before cleaning the inlet, replace dirty column liners and inserts with clean ones. If changing them does not correct the problems, then clean the inlet.

Materials needed:

- Cleaning brushes—The FID cleaning kit contains appropriate brushes (part no. 9301-0985)
- Solvent that will clean the type of deposits in your inlet
- Compressed, filtered, dry air or nitrogen

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Turn the heated zones off—wait for them to cool.
   - Turn off all flows to the inlet at the initial gas supply.
   - Turn off the GC and unplug it.
   - Remove the inlet liner.
   - Remove the column adapter. See "Columns and Traps".
   - Remove the inlet base seal. See page 313 for instructions.
2. Illuminate the inside of the inlet from below and look for signs of contamination or deposits. Insert the brush into the inlet. Scrub the interior walls of the inlet vigorously to remove all deposits.
3. Blow out loose particles and dry thoroughly with compressed air or nitrogen before reassembling.
4. Reassemble the inlet. Use a new inlet base seal. Restore to normal operating conditions.

15 The Purged Packed Inlet

Using a Purged Packed Inlet

Liners and inserts

Procedure: Installing liners
Procedure: Installing glass inserts

Columns and Traps

The control table

Packed columns or column not defined
Defined capillary columns
Procedure: Using packed and undefined capillary columns
Procedure: Using defined capillary columns

Maintaining a Purged Packed Inlet

Procedure: Changing septa
Procedure: Changing the O-ring
Procedure: Leak testing the gas plumbing
Procedure: Leak testing an EPC purged packed inlet
Procedure: Leak testing a nonEPC purged packed inlet
Procedure: Correcting leaks
Procedure: Cleaning the inlet

The Purged Packed Inlet

Using a Purged Packed Inlet

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

If a capillary column is used and the column is defined, the inlet is pressure-controlled. If the column is not defined (packed columns and undefined capillary columns), the inlet is flow-controlled.

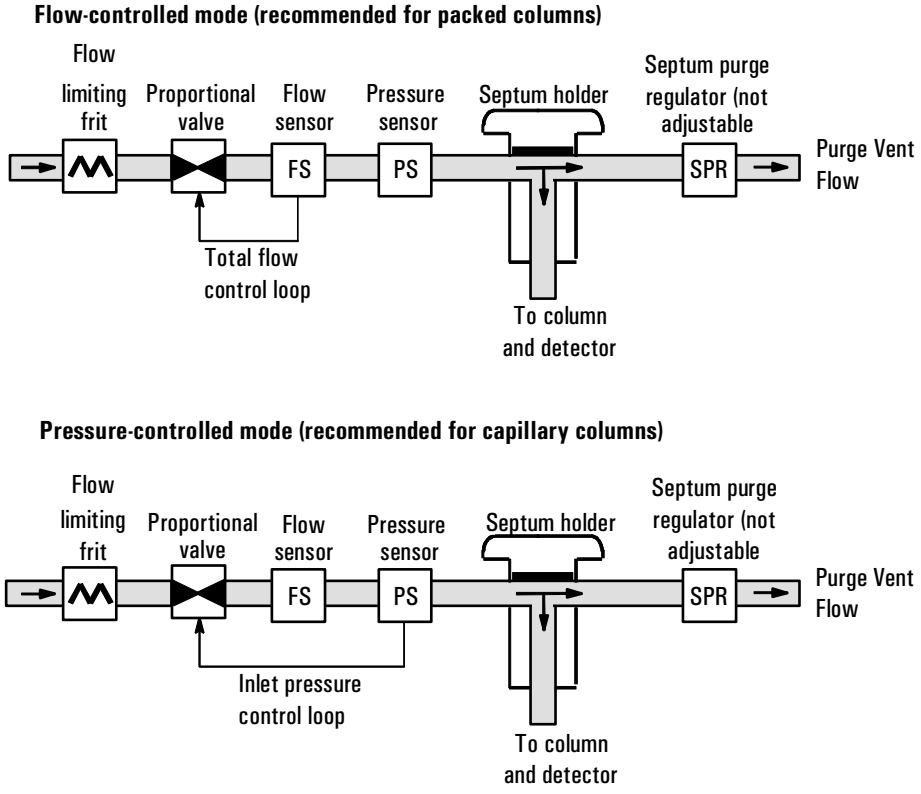


Figure 48 Packed column inlet with electronic pneumatics control

Liners and inserts

Liners. Your choice of liner depends on the type of column you are using. Liners are available for use with wide-bore capillary, 1/4-inch packed, or 1/8-inch packed columns. The liner functions as an adapter so that columns can be connected to the inlet. Installation instructions are on page 327.

Inserts. Glass inserts are often used with metal liners to reduce reactivity and trap nonvolatile residues. They are always used with capillary columns. Inserts are installed from the top of the inlet and should be installed before the column. Installation instructions are on page 329.

The purged packed inlet is shipped with a liner and insert for use with capillary columns; see Table 36. Note that narrow-bore capillary columns are not recommended for use with this inlet. If you are using packed columns, consult Table 37.

Table 36. Liner and Insert for Wide-Bore Capillary Columns

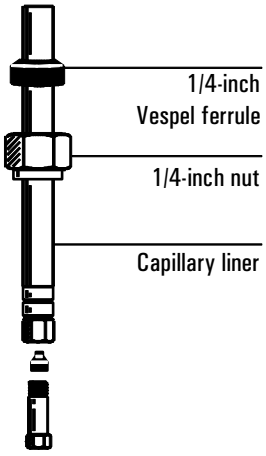
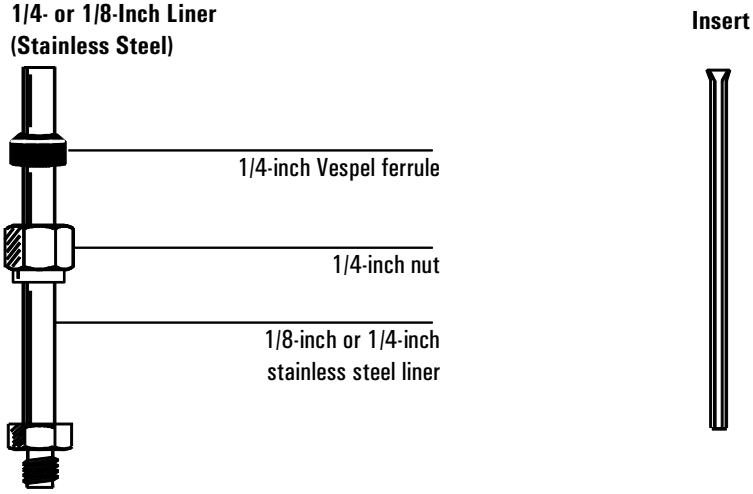
Column type	Liner	Insert
530 µm or 320 µm	19244-80540 	5080-8732 or 5181-3382 (deactivated) 

Table 37 Liner and Insert for Packed Columns

Column type	Liner	Insert
1/8-inch metal	*1/8-inch stainless steel* 19243-80510	None
	19243-80530	5080-8732 or 5181-3382*
1/4-inch metal	*1/4-inch stainless steel* 19243-80520	None
	19243-80540	5080-8732 or 5181-3382*
1/4-inch glass	No liner required. Column end functions as liner. Can also use 1/4-inch metal liner.	Not applicable

*Deactivated



Procedure: Installing liners

Use these instructions for installing all liner types. Graphitized Vespel ferrules are recommended because metal ferrules tend to lock permanently onto the liner. If a leak develops when using metal ferrules, you must replace the entire liner.

Materials needed:

- Liner, brass nut, and ferrule (see Table 36 or Table 37)
- Lint-free cloth
- Methanol
- 9/16-inch wrench

1. Press [Oven] and set the oven to 35°C. When the temperature reaches setpoint, turn the oven off. Press [Front Inlet] or [Back Inlet] and turn off the inlet temperature and pressure or flow.

WARNING Be careful. The oven and inlet fittings may be hot enough to cause burns.

2. Clean the end of the liner with a lint-free cloth to remove contamination such as fingerprints. Use methanol as a solvent.
3. Place a brass nut and graphitized Vespel ferrule on the liner.
4. Open the oven door and locate the inlet base. Insert the liner straight into the inlet base as far as possible.
5. Hold the liner in this position and tighten the nut finger tight.
6. Use a wrench to tighten the nut an additional 1/4 turn.
7. Install the column.
8. Establish a flow of carrier gas through the inlet, and heat the oven and inlet to operating temperatures. Allow these to cool, and then retighten the fittings.

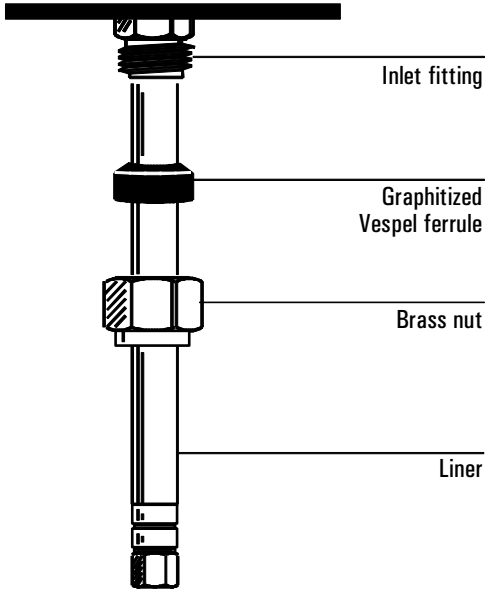


Figure 49 Installing a liner

Procedure: Installing glass inserts

Materials needed:

- Insert (see Table 36 or Table 37)
- Tweezers or hemostats
- Wire

1. Press [Oven] and set the oven to 35°C. When the temperature reaches setpoint, turn the oven off. Press [Front Inlet] or [Back Inlet] and turn off the inlet temperature and pressure or flow.

WARNING Be careful. The inlet fittings may be hot enough to cause burns.

2. Remove the knurled nut at the top of the inlet.
3. Carefully remove the old insert. A thin wire (such as a paper clip) may be helpful when lifting the insert from the inlet.

4. Using tweezers or similar tool, grasp the top of the insert and install in the inlet with the flared end up.
5. If a capillary column is installed and the insert does not seat properly, you must remove the capillary column, install the insert, and replace the column.
6. Reinstall the knurled nut and tighten finger tight.

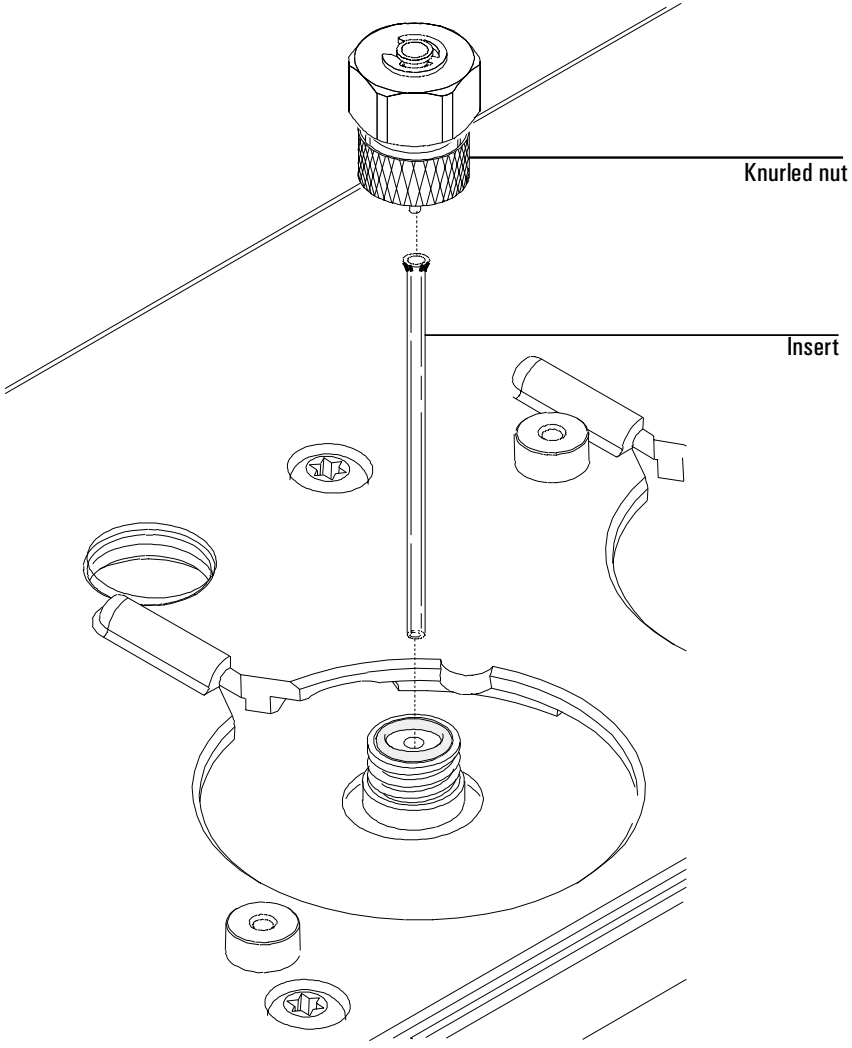
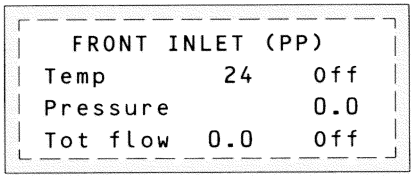


Figure 50 Installing a glass insert in a purged packed inlet

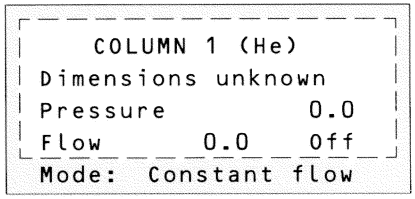
The control table

Packed columns or column not defined

(The inlet)



(The column)



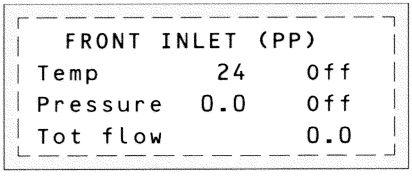
`Temp` The setpoint and actual temperature values.

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

`Tot flow` Enter your setpoint here, actual value is displayed. Inlet is mass flow controlled.

Defined capillary columns

(column defined)



`Temp` The setpoint and actual temperature values

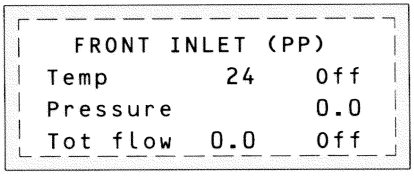
`Pressure` Inlet is pressure controlled. Enter your setpoint here (in psi, bar, or kPa) and actual value is displayed.

`Tot flow` The actual total flow to the inlet. This is a reported value, not a setpoint.

Procedure: Using packed and undefined capillary columns

If the column is not defined, only the flow-controlled modes are available.

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet] or [Back Inlet] and enter a temperature. (The flow was set at the column in step 4.)

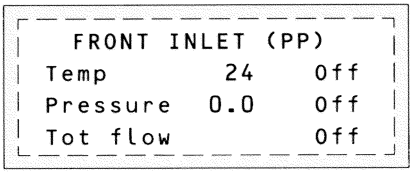


3. Inject a sample.

Set column flow from the Column table. Total flow in the inlet table is the sum of column flow and septum purge flow.

Procedure: Using defined capillary columns

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet] or [Back Inlet] and enter a temperature.



3. Inject the sample.

Maintaining a Purged Packed Inlet

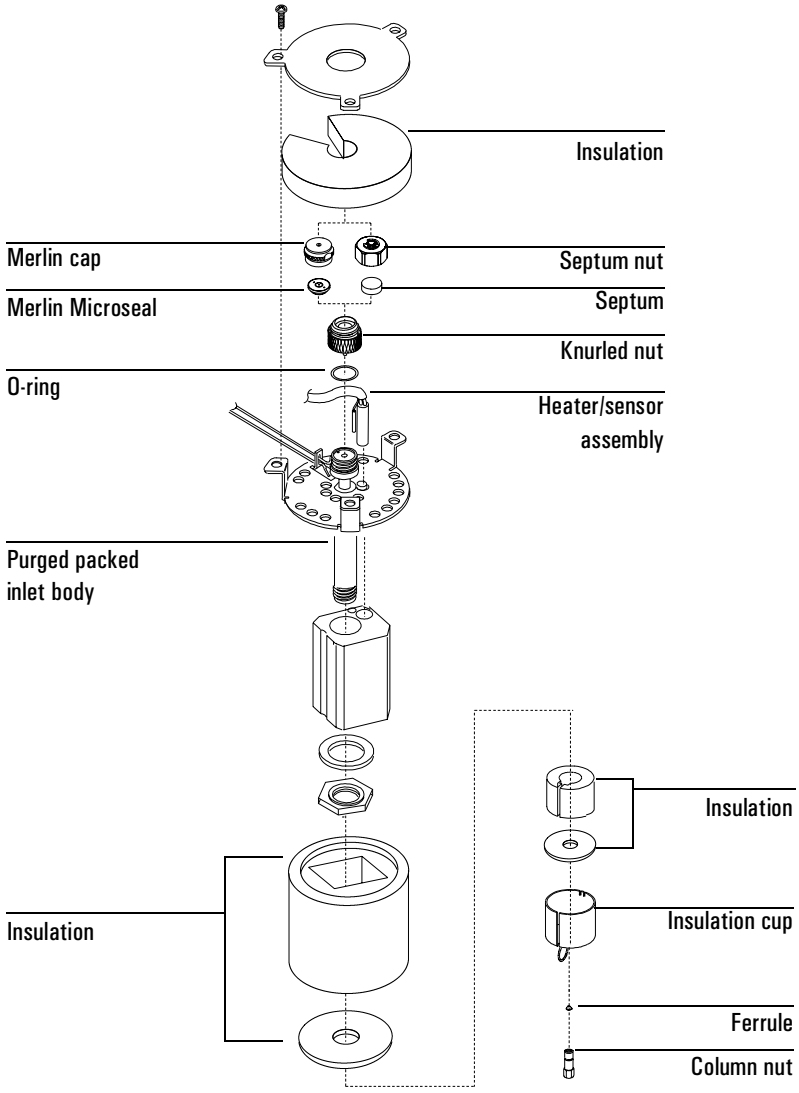


Figure 51 The purged packed inlet

Procedure: Changing septa

If the septum leaks, you will see symptoms such as longer or shifting retention times, loss of response, and/or loss of column head pressure. Additionally, the detector signal will become increasingly noisy.

The useful lifetime of septa is determined by injection frequency and needle quality; burrs, sharp edges, rough surfaces, or a blunt end on the needle decrease septum lifetime. When the instrument is used regularly, daily septum replacement is recommended.

The type of septa you use will depend on your chromatography needs. Another available option is the Merlin Microseal septum, a duckbill setup providing low bleed and long life when used with the 7683 Automatic Liquid Sampler and recommended syringes. You can order septa directly from Agilent Technologies; see the Agilent catalog for consumables and supplies for ordering information.

Table 38. Recommended Septa for the Purged Packed Inlet

Description	Part no.
11-mm septum, low-bleed red	5181-1263
11-mm septum with partial through-hole, low-bleed red	5181-3383
11-mm septum, low-bleed gray	5080-8896
Merlin Microseal septum (30 psi)	5181-8815
11-mm high-temperature silicon septum (350° C and higher)	5182-0739

WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

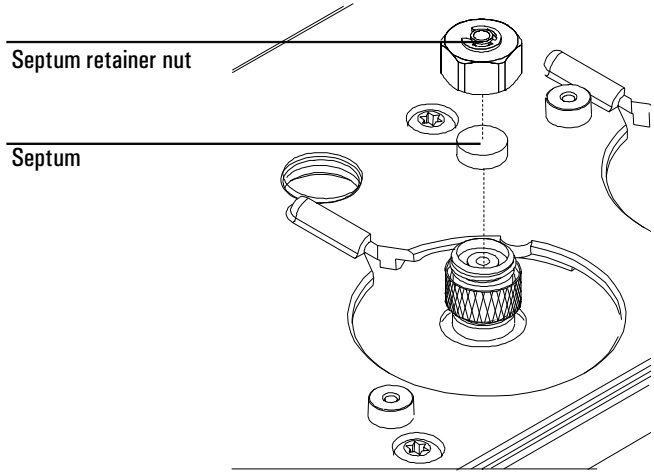
Caution Column flow is interrupted while changing septa; since columns may be damaged at elevated temperatures without carrier flow, cool the oven to room temperature before proceeding.

Materials needed:

- Gloves (if the inlet is hot)
- New septum—see Table 38 for part numbers
- Septum nut wrench (part no. 19251-00100)
- A plastic or wood tool with a sharp tip to remove septum from inlet
- 0- or 00-grade steel wool (optional)
- Forceps or tweezers
- Compressed, filtered, dry air or nitrogen (optional)

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Turn the oven off and let it cool to room temperature.
   - Turn off the detector.
   - Cool the inlet to room temperature.
   - Turn the inlet pressure off.

2. If the inlet is hot, wear gloves to protect your hands from burns. Remove the septum retainer nut or Merlin cap, using the wrench to loosen or remove the nut if it is hot or sticks. Remove the old septum or Merlin Microseal.

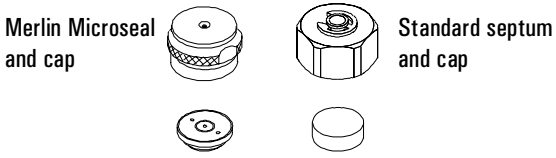


   If the septum sticks, use the sharp-tipped tool to remove it. Take care not to gouge the metal around the septum and to remove all pieces of the old septum.

3. If pieces of the septum are sticking, grasp a small piece of steel wool with the forceps or tweezers and scrub the residue from the retainer nut and septum holder. Use compressed air or nitrogen to blow away the pieces of steel wool and septum.

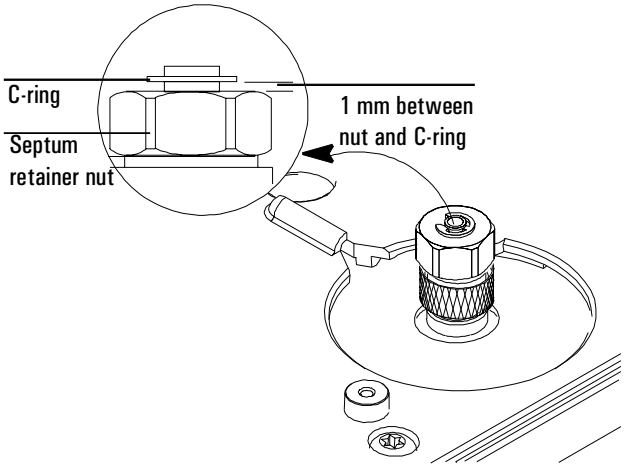
   Use the forceps to insert a new septum or Merlin Microseal. Press it into the fitting firmly.

4. If installing a Merlin Microseal, install it so that the side with the metal parts faces down (toward the oven).



5. Replace the septum retainer nut or Merlin cap.

- If using the standard septum retainer nut, tighten until until the C-ring is approximately 1 mm above the nut. Avoid overtightening.
- If using a Merlin cap, finger tighten until snug (not loose).



Do not overtighten the nut. Over-tightening over-compresses the septum, which could cause premature failure and pieces of septum to contaminate the inlet.

6. Restore normal operating conditions.

Procedure: Changing the O-ring

You will need to change the O-ring periodically because it wears out and becomes a source of leaks in the inlet. To determine if the O-ring leaks, perform the leak test presented in "Procedure: Leak testing an EPC purged packed inlet".

O-rings contain plasticizers that give them elasticity. The O-ring seals the top of the inlet and the inlet base. However, at high temperatures the plasticizers bake out, and the O-rings become hard and are unable to create a seal. If you operate the inlet at high temperatures, you will probably need to replace the O-ring frequently.

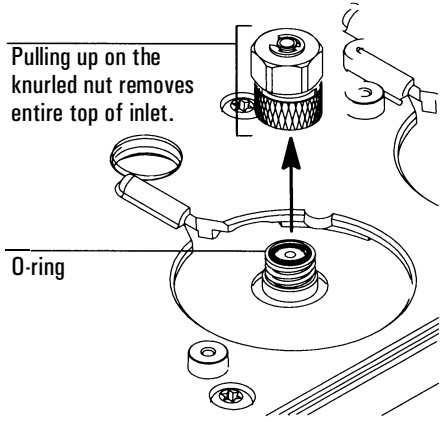
WARNING Be careful! The oven and/or inlet may be hot enough to cause burns. If the inlet is hot, be sure to wear gloves to protect your hands.

Materials needed:

- Gloves (if the inlet is hot)
- A new Viton O-ring (part no. 5080-8898)
- Septum nut wrench (part no. 19251-00100)
- Forceps or tweezers (optional)

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Turn the oven off and let it cool to room temperature.
   - Turn off the detector.
   - Cool the inlet to room temperature.
   - Turn the inlet pressure off.
2. If the inlet is hot, use the septum nut wrench. Loosen the knurled nut completely. Pull up on the nut to remove the top portion of the inlet.

   The O-ring will be visible. Remove the old O-ring. You may need to use forceps to grab it. Using the tweezers, insert the new O-ring.



3. Replace the top portion of the inlet and tighten the knurled nut until you cannot tighten it further. Restore the GC to normal operating conditions.

Procedure: Leak testing the gas plumbing

Leaks in the gas plumbing system can affect chromatographic results dramatically. The following procedure checks the flow system up to but not including the inlet flow manifold. If this portion of the system proves to be leak-free, refer to the next procedure to check the inlet and inlet manifold.

Liquid leak detectors are not recommended, especially in areas where cleanliness is very important.

If you do use leak detection fluid, immediately rinse the fluid off to remove the soapy film.

WARNING To avoid a potential shock hazard when using liquid detection fluid, turn the GC off and disconnect the main power cord. Be careful not to spill leak solution on electrical leads, especially the detector heater leads.

Materials needed:

- Electronic leak detector or liquid leak detection fluid. If you use leak detection fluid, wipe off excess fluid when you have completed the test.
- Two 7/16-inch wrenches

1. Using the leak detector, check each connection you have made for leaks.
2. Correct leaks by tightening the connections. Retest the connections; continue tightening until all connections are leak-free.

Procedure: Leak testing an EPC purged packed inlet

This procedure allows you to determine if the inlet is leaking. It is recommended that you leak test the inlet at your normal operating temperature since the O-ring may leak if it is cooled to ambient.

Materials needed:

- Gloves (if the inlet is hot)
- Septum nut wrench (part no. 19251-00100)
- 1/8-inch SWAGELOK cap (part no. 5180-4120)

If you are using capillary columns:

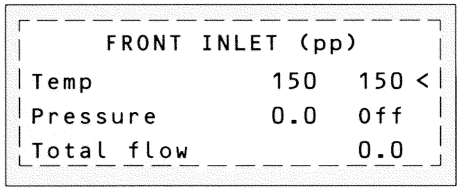
- No-hole ferrule
- 7/16-inch wrench

If you are using packed columns:

- Solid Vespel plug
- 9/16-inch wrench

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Turn the oven off and let it cool to room temperature. When the oven is cool, turn off the inlet pressure.
   - Remove the column, if one is installed, and cap the column fitting. If you are using capillary columns, insert a no-hole ferrule in the column nut to create a plug. If you are using packed columns, use the Vespel plug.
   - Remove the old septum and replace it with a new one. For instructions on changing septa, see "Procedure: Changing septa".
   - Inspect the O-ring and replace it if it is hard and brittle or cracked. See page 337 for instructions on changing the O-ring.
   - Make sure that the pressure at the gas source is at least 35 psi.
   - Cap the septum purge fitting with a 1/8-inch SWAGELOK cap.
   - Define a capillary column to put the inlet into pressure control mode. Press [Column 1] or [Column 2], and enter any diameter (e.g., 320) and length 0. Press [enter].

2. Press [Front Inlet] or [Back Inlet] to open the control table.



3. Set the inlet to its normal operating temperature.
4. Set the inlet pressure to 25 psi. Wait a few minutes for the pressure to equilibrate. The pressure may exceed the setpoint briefly while it equilibrates. If it cannot reach setpoint, either there is a large leak or the gas supply pressure is too low.
5. Turn the inlet pressure `Off`. Because the column is capped, the pressure should remain fairly constant.

   Monitor the pressure for 10 minutes. A pressure drop of 0.3 psi(0.03 psi/min or less) is acceptable. If the pressure drop is much greater than 0.7 psi, go to “Correcting Leaks” on page 343.

Procedure: Leak testing a nonEPC purged packed inlet

This procedure allows you to determine if the inlet leaks. It is recommended that you leak test the inlet at your normal operating temperature since the O-ring is likely to leak if it is cooled to ambient.

Materials needed:

- Gloves (if the inlet is hot)
- Septum nut wrench (part no. 19251-00100)
- 1/8 inch SWAGELOK cap (part no. 5180-4120)

If you are using capillary columns:

- No-hole ferrule
- 7/16-inch wrench

If you are using packed columns:

- Solid Vespel plug
- 9/16-inch wrench

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Turn the oven off and let it cool to room temperature. When the oven is cool, turn off the inlet pressure.
   - Remove the column, if present, and cap the column fitting. If you are using capillary columns, insert a no-hole ferrule in the column nut to create a plug. If you are using packed columns, use the Vespel plug.
   - Remove the old septum and replace it with a new one. For instructions on changing septa, see "Changing septa".
   - Inspect the O-ring and replace it if it is hard and brittle or cracked. See "Changing the O-ring" for instructions on changing the O-ring.
   - Make sure that the pressure at the gas source is at least 30 psi.
2. Set the inlet to its normal operating temperatures.
3. Cap the septum purge vent with a 1/8-inch SWAGELOK cap.
4. Turn on the gas to the inlet at its source and adjust the supply pressure to 30 psi. Completely open the mass flow controller by turning the knob counterclockwise as far as it can go. Wait 2 minutes to insure equilibrium. The gauge or the front panel should be stable.
5. Shut off the column head pressure by turning the flow controller full clockwise. Do not overtighten or you will damage the valve seat.
6. Turn off the gas to the inlet at its source. Monitor the pressure for 10 minutes. You can use the GC's Stopwatch function. A pressure drop of 0.7 psig (0.07psi/min or less) is acceptable.

If the pressure drop is 0.7 psi (0.07 psi/min) or less, you can consider the inlet leak-free.

If the pressure drop is much greater than 0.7 psi (0.07 psi/min) go to "Procedure: Correcting leaks".

Procedure: Correcting leaks

Materials needed:

- Electronic leak detector suitable for the gas type
- Tools to tighten parts of the inlet that leak (if leaks are detected)

1. Use the leak detector to check all areas of the inlet that are potential sources of a leak. Potential leak areas are:

   - The septum and/or septum nut
   - The 1/4-inch ferrule (if a liner is being used)
   - The O-ring
   - The capped purge vent
   - The plugged column connection
   - The knurled nut
   - The area where the gas line is plumbed to the inlet

   If no liner is used, then column must be plugged with 1/4-inch SWAGELOK cap or equivalent.

2. Correct leaks using a wrench to tighten loose connections, if necessary. You may need to repeat the leak test.

   If the pressure drop is now 0.03 psi/m inch or less, you can consider the inlet leak-free.

   If the pressure drops faster than the acceptable rate, continue to search for leaks and repeat the pressure test. If all fittings appear to be leak free, but the inlet is still losing too much pressure, you may need to replace the inlet manifold. Contact your Agilent service representative.

Procedure: Cleaning the inlet

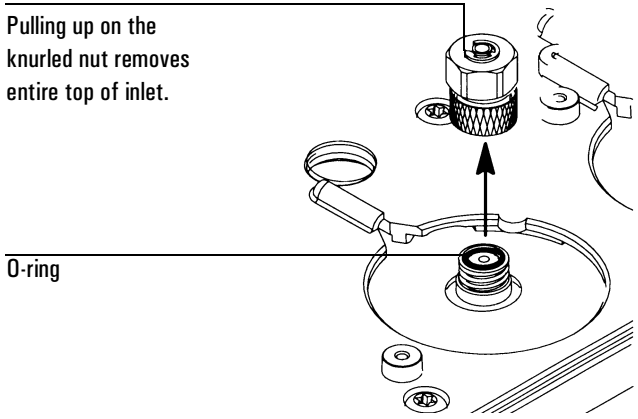
It is unlikely that the inlet will frequently require cleaning as thoroughly as this procedure presents; however, deposits from injected samples occasionally build up inside the purged packed inlet. Before cleaning the inlet, replace dirty column liners and inserts with clean ones. See "Procedure: Installing liners", "Procedure: Installing glass inserts" for instructions. If changing them does not correct the problems, then clean the inlet.

Materials needed:

- Cleaning brushes—The FID cleaning kit contains appropriate brushes (part no. 9301-0985)
- Solvent that will clean the type of deposits in your inlet
- Compressed, filtered, dry air or nitrogen

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Allow the heated zones to cool.
   - Turn off all flows to the inlet at the initial gas supply.
   - Turn off the GC and unplug it.
   - If the septum is worn out or dirty, replace it. See "Procedure: Changing septa" for instructions.
   - Remove the column and the column liner and insert. See "Columns and Traps".

2. Loosen the knurled nut and pull it upward. The O-ring will be visible. Replace it if it is hard and brittle or cracked. See "Procedure: Changing the O-ring" for the procedure.



3. Use a light source to illuminate the inside of the inlet from inside the oven while looking through the inlet from the top. If deposits are present, they should be visible.
4. Insert the brush into the inlet. Scrub the interior walls of the inlet vigorously to remove all deposits. You may need to wet the brush with solvent. Use a burst of compressed air or nitrogen to dry the inlet and remove loose contaminants.
5. Replace the top of the inlet and tighten the knurled nut. Replace the column (see "Procedure: Installing glass packed columns").
6. Restore the GC to normal operating conditions.

16 The Cool On-Column Inlet

Using a Cool On-Column Inlet

Hardware

Columns and Traps

Automatic or manual injection with septum nut

Septum nuts

Septa

Manual injection with a cooling tower and duckbill septum

Procedure: Changing the septum nut or cooling tower and septum

Procedure: Installing an insert

Procedure: Check the needle-to-column size

Procedure: Manual injection with septum nut

Procedure: Manual injection with cooling tower

Retention gaps

Inlet temperature

CryoBlast (optional)

Track oven mode

Temperature programming mode

Cryogenic considerations

Setpoint ranges

Procedure: Programming the temperature

Procedure: Operating the cool on-column inlet

Maintaining a Cool On-Column Inlet

Cool on-column inlet hardware problems

The inlet cools very slowly

The inlet is unable to reach a temperature setpoint

The syringe needle bends during injections

Procedure: Replacing the fused silica syringe needle

Procedure: Installing a fused silica needle

Changing septa

Procedure: Changing septa

Procedure: Cleaning the inlet

Procedure: Leak testing the gas plumbing

Procedure: Leak testing a cool on-column inlet

Procedure: Correcting leaks

The Cool On-Column Inlet

Using a Cool On-Column Inlet

This inlet introduces liquid sample directly onto a capillary column. To do this, both the inlet and the oven must be cool at injection, at or below the boiling point of the solvent. Because the sample does not vaporize immediately in the inlet, problems with sample discrimination and sample alteration are minimized. If done properly, cool-on column injection also provides accurate and precise results.

You can operate the inlet in track oven mode, where the inlet temperature follows the column oven, or you can program up to three temperature ramps. There is also a cryogenic cooling option that uses liquid CO_2 or N_2 to reach sub-ambient temperatures.

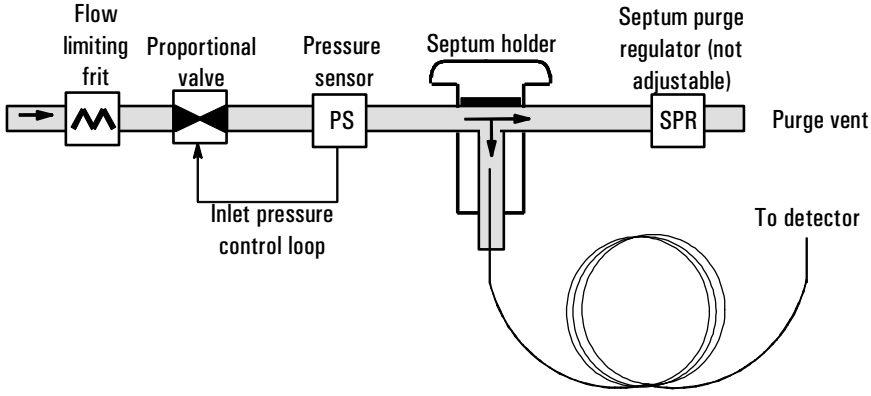


Figure 52 Cool on-column capillary inlet with EPC

Hardware

Because you are injecting sample directly into the column, most of the hardware required is determined by your column inside diameter. Injection technique, manual or automatic, must also be considered. Table 39 is a checklist for choosing hardware and shows where to find instructions for installing the hardware and injecting the sample.

Note that if you are performing automatic injections on a 250 µm/320 µm column using a 7683 ALS, you must adapt your autosampler for on-column use. Refer to the manual(s) listed in Table 39 below.

Table 39. Hardware and Procedures Checklist

Automatic injection	Manual injection with septum nut	Manual injection with cooling tower
Hardware		
See Table 40 for part numbers	See Table 40 for part numbers	See Table 41 for part numbers
❒ Septum nut ❒ Insert ❒ Stainless steel needle	❒ Septum nut ❒ Solid septum ❒ Insert ❒ Stainless steel needle	❒ Cooling tower ❒ Duckbill septum ❒ Insert ❒ Fused silica needle (columns ≥200 µm) or ❒ Stainless steel needle (columns ≥250 µm)
Where to find instructions		
❒ Installing an Insert, page 353 ❒ Changing the septum nut or cooling tower assembly, page 352	❒ Installing an Insert, page 353 ❒ Changing the septum nut or cooling tower assembly, page 352	❒ Installing an Insert, page 353 ❒ Changing the septum nut or cooling tower assembly, page 352
❒ Checking the needle-to-column size, page 354 ❒ 7683 Automatic Liquid Sampler Installation guide, part no. G2613-90107 ❒ 7683 Automatic Liquid Sampler Operation Guide, part no. G2612-90117	❒ Manual injection technique with septum nut and stainless steel needle, page 355 (top)	❒ Manual injection technique with cooling tower, page 355 (bottom) *and* Replacing the fused silica syringe needle, page 363

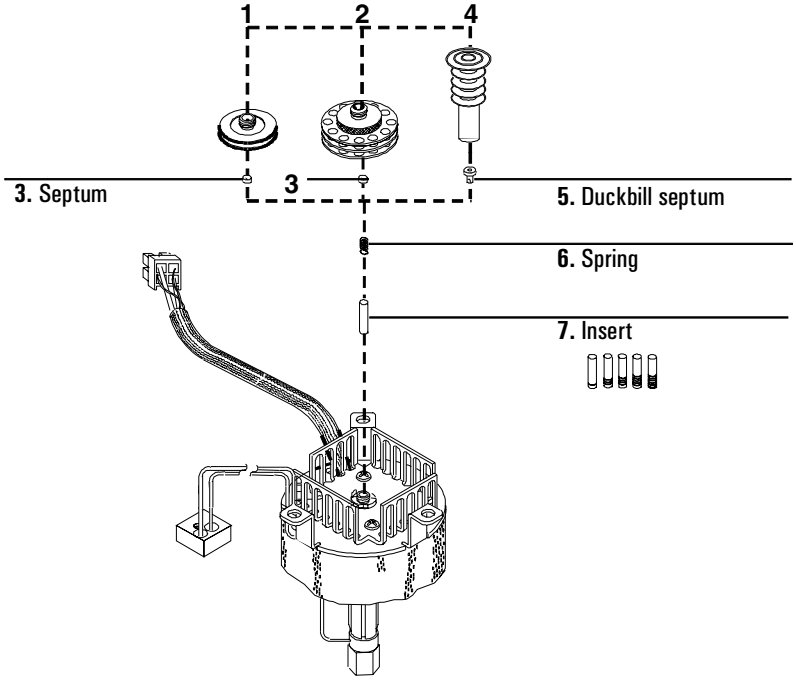


Figure 53 Hardware for the cool on-column inlet

Septum nut and septum, manual or automatic injection

1. Septum nut (part no. 19245-80521) for use with 250-µm and 320-µm columns. See Sampler manual for needle support assembly requirements.
2. Septum nut (part no. G1545-80520) for use with 530-µm columns
3. Septum

Cooling tower and duckbill septum, manual injection

4. Cooling tower assembly (part no. 19320-80625)
5. Duckbill septum (part no. 19245-40050) for columns 200 µm and larger

For all applications:

6. Spring. Keeps insert in position.

7. Insert. Guides the needle into the column. Choose based on column and needle. See Table 40 and Table 41.

Automatic or manual injection with septum nut

Choose a needle, septum nut, and insert based on your column inside diameter. Use Table 40 to select hardware for your injection. See Table 41 if you are doing manual injections with a duckbill septum.

Septum nuts

19245-80521

G1545-80520

Table 40. Automatic or Manual Injection with a Stainless Steel Needle

Column type and inside diameter	Needle part no.*	Septum nut part no.	Insert part no.
Fused silica:			
530 µm id	5182-0832**	G1545-80520	19245-20580 (no rings)
320 µm id	5182-0831	19245-80521	19245-20525 (5 rings)
250 µm id	5182-0833	19245-80521	19245-20515 (6 rings)
200 µm id	Use cooling tower and duckbill septum		19245-20510 (1 ring)
Aluminum-clad, 530 µm id	5182-0832	G1545-80520	19245-20780 (4 rings)
Glass capillary			
320 µm id	5182-0831	19245-20670	19245-20550 (3 rings)
250 µm id	5182-0833	19245-20670	19245-20550 (3 rings)

* Order removable needle syringe, part no. 5182-0836. If doing a manual injection, you must also order a plunger button, part no. 5181-8866.

** Many other needles can be used to inject onto a 530-µ column. Consult the Agilent catalog for consumables and supplies for details.

Septa

Use a solid septum (5181-1261) for manual injection, or a through-hole septum (5181-1260) for auto injection.

Manual injection with a cooling tower and duckbill septum

If you are doing this type of manual injection, use either fused silica or metal removable stainless steel needles. Use Table 41 to choose the correct insert and syringe.

Table 41. Manual Injection Hardware—Cooling Tower & Duckbill Septum

Column type and inside diameter	Insert (part no.)
Fused silica	
530 µm	19245-20580 (no rings)
320 µm	19245-20525 (5 rings)
250 µm	19245-20515 (6 rings)
200 µm	19245-20510 (1 ring)
Aluminum-clad, 530 µm	19245-20780 (4 rings)
Glass capillary	19245-20550 (3 rings)
Syringe and needle	
For fused silica needles	
Fused silica needle syringe	9301-0658
Replacement needles, fused silica, 0.18 mm (6 pk)	19091-63000
Replacement Teflon® ferrule for syringe	0100-1389
For stainless steel needles	
Removable needle syringe, 10 µL	5182-9633
Replacement needles, 0.23 mm (3 pk)	5182-9645

Procedure: Changing the septum nut or cooling tower and septum

If you need to change the insert, refer also to the next section, "Procedure: Installing an insert"

1. Press [Oven] and set the oven to 35°C. When the temperature reaches setpoint, turn the oven off. Press [Front Inlet] or [Back Inlet] and turn off the inlet temperature and pressure.

WARNING Be careful! The inlet fittings may be hot enough to cause burns.

2. Locate the septum nut or cooling tower assembly at the top of the inlet and remove (see Figure 53). If you are using a cooling tower, grasp the three rings and unscrew. If you are using a septum nut, grasp the knurling and unscrew.

   There should be a small spring at the inlet base. If the spring is stuck to the septum nut, place it back in the inlet base.

3. If you are using a *septum nut*, remove the old septum with tweezers, hemostat, or septum remover. Use tweezers to install a new septum. Push the septum into the septum nut until properly seated.

   If you are using a *cooling tower assembly*, locate the duckbill septum and install in the inlet base so that the duckbill is inserted inside the coil spring.

4. Install the septum nut or cooling tower assembly and tighten firmly.

5. Before making an injection, check the alignment of the entire assembly.

Procedure: Installing an insert

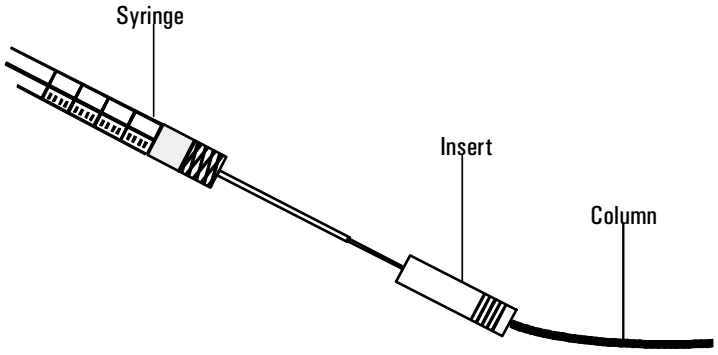
1. Choose an insert. See Table 40 or Table 41 for instructions on choosing an insert.
2. Press [Oven] and set the oven to 35°C. When the temperature reaches setpoint, turn the oven off. Press [Front Inlet] or [Back Inlet] and turn off the inlet temperature and pressure.
3. Remove the column, column nut, and ferrule.
4. Locate the septum nut or cooling tower assembly at the top of the inlet and remove it. If the septum remains in the septum nut, do not remove it unless you want to change it. If necessary, replace the existing septum or duckbill with a new one. See "Maintaining a Cool On-Column Inlet" for detailed instructions. Set the inlet septum nut or cooling tower assembly aside.
5. Remove the spring from the inlet with an extraction wire, and set it aside. Be careful not to lose or damage it because you will use the spring to keep the new insert in position.
6. Remove the existing insert from the inlet by gently pushing it out from below with a wire or piece of column. Store the insert for possible later use.
7. Drop the new insert straight into the inlet from the top.
8. Replace the spring on top of the insert.
9. Reinstall the septum nut or duckbill septum and cooling tower assembly and tighten finger tight.
10. Reinstall the column, nut, and ferrule.

Procedure: Check the needle-to-column size

Caution This applies to 250 µm and 320 µm columns only.

After selecting an insert and before installing a column, you need to check the needle-to-column size to make certain your needle fits in the column. You could bend the needle if you try to inject it into a smaller column. Use the insert that is the same size as your syringe needle to verify that the column you plan to use is the correct size.

1. Identify the correct insert.
2. Insert the column into one end of the insert as shown below.



3. Insert the syringe needle through the other end of the insert and into the column. If the needle cannot pass easily into the column, reverse the insert to try the needle and column in the other end.

If the needle still cannot pass into the column, you may have a column with an incorrect id. Check the column to make sure it is labeled correctly, and try a new column.

Procedure: Manual injection with septum nut

Before making your injection, make sure the correct septum nut and septum are installed.

1. Immerse the syringe needle in sample; pump the syringe plunger to expel air from the barrel and needle.
2. Draw the sample into the syringe.
3. Remove the needle from the sample and draw about 1 µL of air into the syringe.
4. Wipe the needle dry if it is wet.
5. Guide the needle straight into the septum nut, pierce the septum, and insert the needle fully into the inlet until it bottoms.
6. Start the run, depress the syringe plunger *as quickly as possible*, and withdraw the needle from the inlet.

These steps should be done smoothly, with minimal delay.

Procedure: Manual injection with cooling tower

When injecting with fused silica or metal removable stainless steel needles, be sure the cooling tower assembly and duckbill are installed on the inlet. Initial pressure must be set at less than 30 psi. Higher pressures will make needle insertion difficult.

1. Immerse the syringe needle in the sample and pump the syringe plunger to expel air from the barrel and needle.
2. Draw the sample into the syringe. Allow enough time for fluids to pass through the small bore of the needle.
3. Remove the needle from the sample and draw about 1 µl of air into the syringe. Wipe the needle with a tissue wetted with solvent.
4. Press down the top of the cooling tower with a pencil to open the duckbill.

WARNING The cooling tower may be hot!

5. Hold down the cooling tower and guide the needle until it is fully inserted in the inlet. You may observe a drop in the pressure reading on the control table.

   If the needle does not go in all the way, try rotating the syringe and slightly releasing pressure on the cooling tower.

   If you still cannot get the needle in, the duckbill opening may be stuck. Try removing the duckbill, opening it manually, and reinstalling it.

6. Once the needle has entered the column, release the cooling tower and continue to insert the needle. Allow 1 to 2 seconds for back pressure on the duckbill to seal it around the inserted needle.

7. Start the GC, depress the syringe plunger as quickly as possible, and withdraw the needle from the inlet.

Retention gaps

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

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

Inlet temperature

CryoBlast (optional)

CryoBlast shortens the cycle time between runs. If you have a CO_2 or N_2 cryogenic valve and the CryoBlast feature, you can cool the inlet to –37°C in either the track oven or temperature program modes.

Track oven mode

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

Temperature programming mode

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

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

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

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

Cryogenic considerations

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

Setpoint ranges

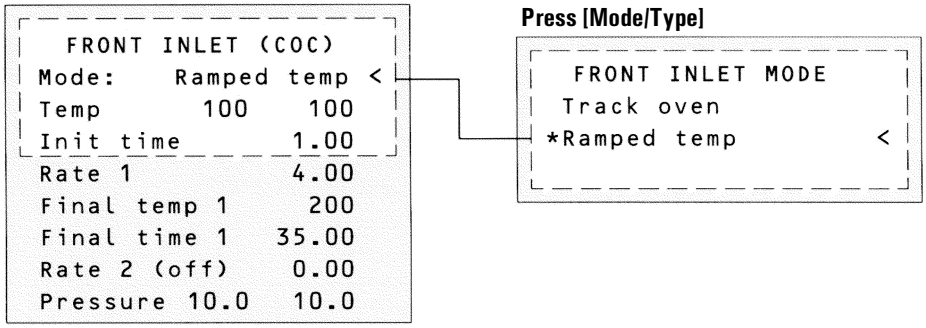
The table below lists setpoint ranges for the inlet parameters.

Temperature	Allowed setpoint range
Track oven	3° C higher than the oven temperature to a maximum of 450° C. If you have CryoBlast, the inlet can maintain temperatures down to −40° C, although allowable oven setpoints are −60° C for CO_2 and −80° C for N_2
Ramped temp *without* CryoBlast	24° C to 450° C
Ramped temp *with* CryoBlast	−40° C to 450° C

Procedure: Programming the temperature

1. Press [Front Inlet] or [Back Inlet].
2. Press [Mode/Type] and select `Ramped temp`.

Ramped temp mode



3. Enter a `Temperature`. This is the starting temperature.
4. Enter an `Init time`. This is the length of time the inlet will stay at the starting temperature after a run has begun.
5. Enter a `Rate`. This is the rate at which the inlet will be heated or cooled. A `Rate` of `0` halts further programming.
6. Enter the `Final temp`. This is the inlet temperature at the end of the first ramp.
7. Enter the `Final time`. This is the number of minutes the inlet holds the `Final temp`.
8. To enter a second (or third) ramp, scroll to the appropriate `Rate` line and repeat steps 5 through 7.

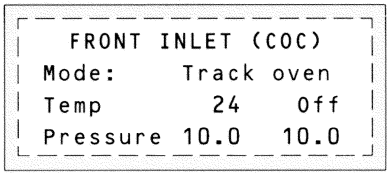
Procedure: Operating the cool on-column inlet

Verify that a column and suitable insert and septum nut or cooling tower are installed. Make certain you are using a needle that will fit the column.

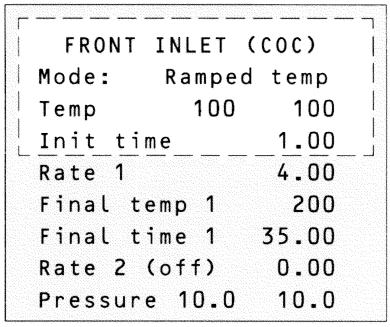
1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control" .

   Pressure can be set from either the column or inlet table. In constant or ramped flow mode, the pressure will be determined from the flow requirements. It is best to set flow only.

Track oven mode



Ramped temp mode



2. Press [Front Inlet] or [Back Inlet]
   a. Choose a temperature mode: `Track oven` or `Ramped temp`.
   b. For `Ramped temp` mode, enter your temperature ramps (page 359). There is no setpoint for `Track oven` mode.
3. Inject a sample.

Maintaining a Cool On-Column Inlet

Maintaining the cool on-column inlet includes changing septa, cleaning inlet components, and checking and correcting leaks in the system.

The cool on-column inlet's hardware will vary depending on whether you will be making manual or automated injections, the type of needle you use, and the size of column you use.

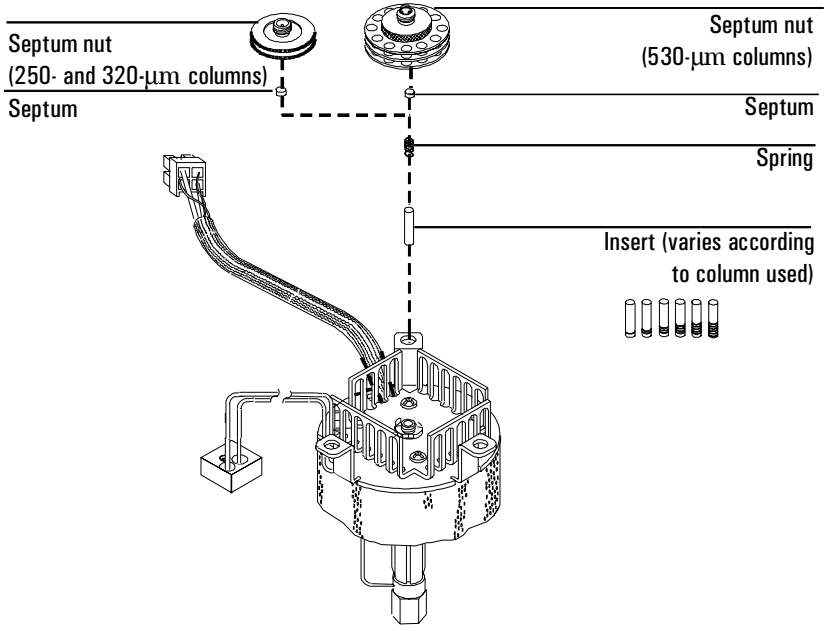


Figure 54 The cool on-column inlet for automatic injection systems

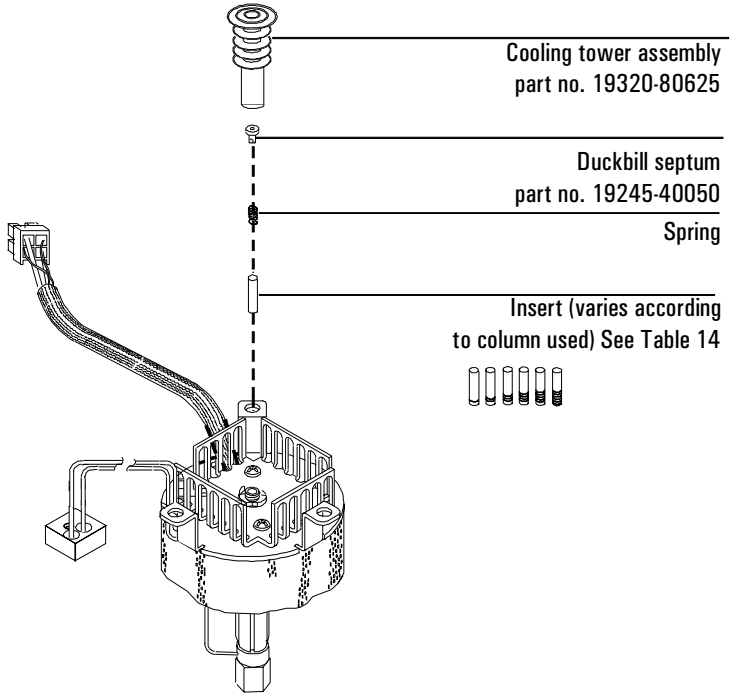


Figure 55 The cool on-column inlet for manual injection systems

Cool on-column inlet hardware problems

The inlet cools very slowly

- The inlet fan is not running or is blowing away from the inlet. Check the fan to make sure it is operating. If it is not, contact your Agilent service representative.

The inlet is unable to reach a temperature setpoint

- Check the temperature equilibration time. If the equilibration time is too short, the inlet may oscillate. Increase the equilibration time.
- Check that cryogenic cooling is turned off. If you do not turn it off when not in use, both the inlet and the oven may be unable to reach their setpoints, particularly temperatures near room temperature. If you turn the cryogenic cooling off and the inlet still fails to reach the setpoint temperature, contact your Agilent service representative.

The syringe needle bends during injections

- The needle may have been defective before the injection was made. Check each syringe before injection to make sure the needle is straight.
- Check that the needle support assembly is installed correctly.
- Check that the correct insert is installed and that it is installed correctly.
- Check the alignment of the inlet septum and the septum nut.
- The inlet septum hole may have closed. Replace the septum.

If you are using the GC Automatic Liquid Sampler (GC ALS):

See the GC ALS manual for additional information.

- The sampler vials may be over-crimped.
- Check the needle guide for signs of wear or damage. Replace the needle guide if necessary.
- Check the alignment of the inlet and the automatic sampler.

Procedure: Replacing the fused silica syringe needle

1. Hold the syringe vertically and insert the fused silica needle so it is visible *inside* the syringe barrel. If the fused silica needle cannot be inserted into the syringe barrel, the Teflon ferrule (part no. 0100-1389) may be blocked.

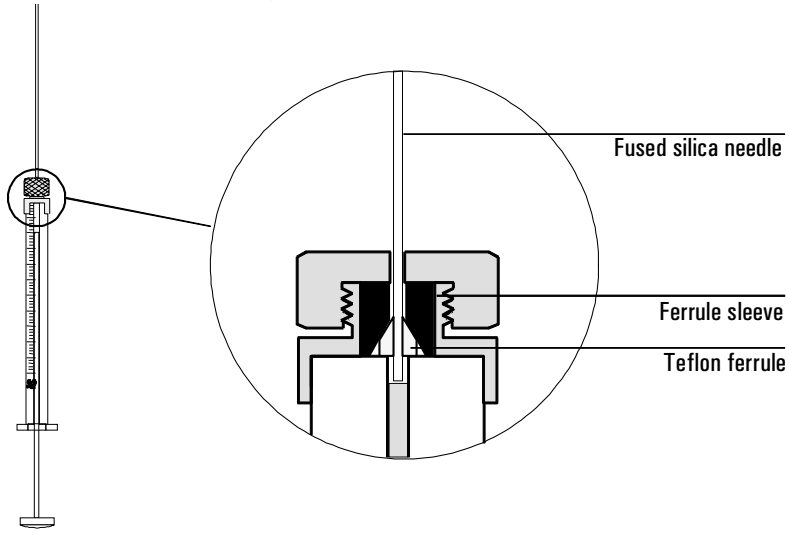
You may need to replace the ferrule. Push the plunger down until it bottoms. The needle will now be flush with the end of the plunger.

2. When the needle is inserted, tighten the retaining nut to *firm* finger tightness. Pull the needle gently to be sure the Teflon ferrule has formed a tight seal with the needle. Tighten the retaining nut further, if necessary.
3. Loosen the retaining nut just enough so the needle is again free. Depress the syringe plunger slowly until it pushes the needle to the end of the barrel, then tighten the retaining nut to *firm* finger tightness.
4. Use a solvent to rinse the syringe and check for leaks or blocks.
5. Leaks (inability to eliminate air bubbles) *may* be fixed by further tightening the retaining nut. Blocks (or serious leaks) require repeating this procedure.

The Teflon ferrule may lose its seal in time. If so, first retighten the retaining nut and, if the seal still leaks, install a new Teflon ferrule and needle.

When not in use, loosen the retaining nut to avoid premature leaks.

Procedure: Installing a fused silica needle



If you are cutting replacement needles directly from fused silica column material:

1. Column material for making needles must have an outside diameter *smaller* than both the inside diameter of the on-column inlet (0.23 mm) and the inside diameter of the installed column.
2. Column material must be washed free of active stationary phase.
3. Score the column material about 1/4-inch from its end. Break off the end and discard. Then measure, score, and break off a 115 $\pm$ 5 mm length to use as the syringe needle.

Changing septa

If the septum leaks, you will see symptoms such as longer or shifting retention times, loss of response, and/or loss of column head pressure. Additionally, the detector signal will become increasingly noisy.

The useful lifetime of septa is determined by injection frequency and needle quality; burrs, sharp edges, rough surfaces, or a blunt end on the needle decrease septum lifetime. When the instrument is used regularly, daily septum replacement is recommended.

The type of septa you use will depend on your chromatography needs. You can order septa directly from Agilent Technologies; see their "Consumables and Accessories Catalog" for ordering information.

Caution The procedure for changing septa differs depending on whether you cool on-column inlet has a cooling tower assembly or a septum nut. Make sure to follow the correct procedure for your inlet!

Table 42. Recommended Septa for the Cool On-Column Inlet

Description	Part no.
Solid septum for manual and automatic injection (50 pk)	5181-1261
Through-hole septum for automatic injection (25 pk)	5181-1260
Solid septum, bleed and temperature optimized (50 pk)	5182-0745
Duckbill septum for manual injection only (must use cooling tower with the duckbill) (10 pk)	19245-40050

WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

Caution Column flow is interrupted while changing septa; since columns may be damaged at elevated temperatures without carrier flow, cool the oven to room temperature before proceeding.

Procedure: Changing septa

Materials needed:

- New septum—see Table 42 for part numbers
- Forceps (or tweezers)
- A thin wire (0.2-inch diameter) for removing septum from inlet

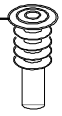
1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Cool the oven to room temperature and then turn the oven off.
   - Cool the inlet to room temperature and then turn the inlet off.

Depending on your analysis and injection technique, the inlet will have one of the following septum nuts or a cooling tower assembly.

Septum nut for injections onto 250- and 320-µm columns

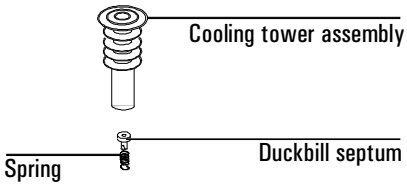
Septum nut for injections onto 530-µm columns

Cooling tower assembly (for manual injections only)



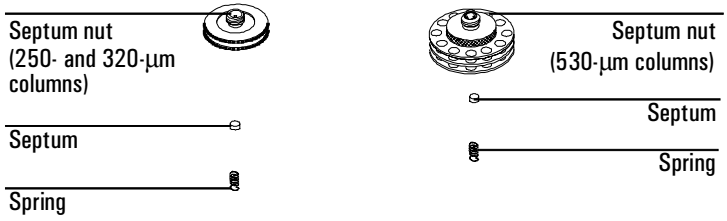
2. **If you have a cooling tower assembly installed:**
   Remove the assembly by grasping it and turning counterclockwise. The duckbill septum is underneath the cooling tower inside the spring. The spring and septum may pop out of the inlet when you remove the cooling tower.

Be careful not to lose them. If they do not pop out, use a thin wire to remove them from the inlet.



Insert the duckbill septum into the spring and place them in the inlet. Reattach the cooling tower assembly. Tighten it finger-tight.

3. **If you have a septum nut installed:**
   Remove the septum nut by grasping the knurling and turning counter-clockwise. The septum is probably attached to the septum nut. The spring may also pop out when you remove the septum nut. Be careful not to lose it. If the septum is not attached, you may need to use tweezers to grasp and remove it.



   Make sure the spring is in the inlet. Use the tweezers to place a new septum on the bottom of the septum nut, and then reattach the septum nut to the inlet. Tighten the nut firmly.

4. Restore normal GC operating conditions.

Procedure: Cleaning the inlet

Most laboratories have airborne lint and dust that accumulate on the cooling tower or septum nut and can enter the inlet or column on the syringe needle. Particulate matter in the inlet interferes with easy passage of the syringe needle. If dirt enters the column, it can alter the chromatography.

You can clean the needle guides, springs and inserts according to the following procedure.

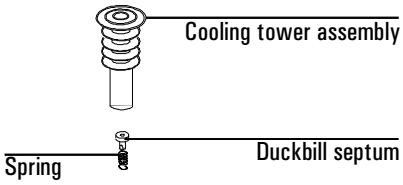
WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

Materials needed:

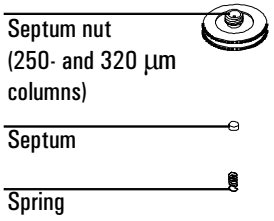
- 9/16-inch wrench
- Narrow wire (0.02-inch diameter) or piece of capillary column (250-µm diameter) for removing spring and insert
- Small ultrasonic cleaning bath with aqueous detergent
- Distilled water
- Methanol
- Compressed, filtered, dry air or nitrogen

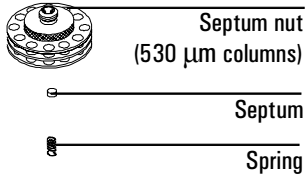
1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Allow the oven and inlet to cool.
   - Turn off all flows to the inlet at the initial gas supply.
   - Turn off the GC and unplug it.
   - Remove the column. See "Procedure: Installing capillary columns in the split/splitless inlet".
2. If you have a cooling tower assembly installed:
   Remove the assembly by grasping it and turning counterclockwise. The septum is underneath the cooling tower inside the spring. The spring and septum may pop out of the inlet when you remove the cooling tower. Be

careful not to lose them. If they do not pop out, use a thin wire to remove them from the inlet.

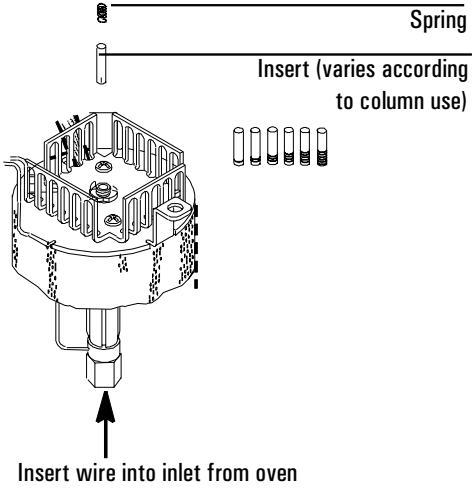


3. If you have a septum nut installed:
   Remove the septum nut by grasping the knurling and turning counter-clockwise. The septum is probably attached to the septum nut. The spring may also pop out when you remove the septum nut. Be careful not to lose it.





4. Insert the narrow wire (or a piece of capillary column) into the inlet through the oven, and push the insert and spring (if they did not come out previously) out through the top of the inlet.



5. Cleaning procedure:
   a. Fill the ultrasonic cleaning bath with aqueous detergent and place the spring and the insert into it. Sonicate for 1 minute.
   b. Drain the aqueous detergent and fill the bath with distilled water. Sonicate for 1 minute.
   c. Remove the parts from the bath and rinse them thoroughly with water and methanol.
   d. Dry the parts with a burst of compressed air or nitrogen.
6. Reinstall the insert. If you are using a septum nut, insert the spring and insert with the spring on top.
7. Attach a new septum to the bottom of the septum nut. If you are using the cooling tower assembly, insert a new duckbill septum into the spring, and place them in the inlet.
8. Attach the septum nut or the cooling tower and tighten finger-tight. Reinstall the column and restore normal operation conditions.

Procedure: Leak testing the gas plumbing

Leaks in the gas plumbing system can affect chromatographic results dramatically. The following procedure checks the flow system up to but not including the inlet flow manifold. If this portion of the system proves to be leak-free, refer to the next procedure to leak-check the inlet and inlet manifold.

Liquid leak detectors are not recommended, especially in areas where cleanliness is very important.

If you do use leak detection fluid, immediately rinse the fluid off to remove the soapy film.

WARNING To avoid a potential shock hazard when using liquid detection fluid, turn the GC off and disconnect the main power cord. Be careful not to spill leak solution on electrical leads, especially the detector heater leads.

Materials needed:

- Electronic leak detector capable of detecting your gas type or liquid leak detection fluid. If you use leak detection fluid, remove excess fluid when you have completed the test.
- Two 7/16-inch wrenches

1. Using the leak detector, check each connection you have made for leaks.
2. Correct leaks by tightening the connections. Retest the connections; continue tightening until all connections are leak-free.
3. Cap the septum purge vent with a 1/8-inch SWAGELOK cap.

Procedure: Leak testing a cool on-column inlet

There are numerous places in the inlet that can leak. This procedure lets you determine, in general, if there is an unacceptable leak in the inlet.
If the inlet is leaking, you should use an electronic leak detector to pinpoint the component that is leaking.

Materials needed:

- No-hole ferrule
- 1/4-inch wrench
- Gloves (if the inlet is hot)

1. Complete the following preliminary steps:
    - If you have entered parameters that you do not want to lose, store them as a method.
    - Allow the oven to cool to room temperature and then turn it off.
    - When the oven is cool, turn off the inlet pressure.
    - Remove the column, if one is installed, and plug the column fitting with the column nut with a no-hole ferrule installed.
    - Remove the old septum and replace it with a new one. For instructions, see page 367.
    - Make sure the carrier gas source pressure is at least 35 psi.

2. Cap the septum purge vent with a 1/8-inch SWAGELOK cap.
3. Press [Oven] to open the control table. Set the oven temperature to its normal operating temperature.
4. Press [Front Inlet] or [Back Inlet].

   Set the inlet to normal operating temperature.

   Enter a pressure to 25 psi, or enter your normal operating pressure if it is higher. Make sure that the pressure at the initial gas supply is at least 10 psi higher than the inlet pressure. If pressure cannot be achieved, either there is a large leak or the gas supply pressure is too low.
5. Wait a few minutes for the GC to equilibrate after the system has reached the pressure. The pressure may exceed the setpoint briefly during equilibration.
6. Turn the pressure off. Because the column is capped, the pressure should remain fairly constant.
7. Monitor the pressure for 10 minutes.
   - A pressure drop of 1.0 psi (0.1 psi/min) or less is acceptable.

   If the pressure drop is much greater than 1.0 psi, go to the next section, "Procedure: Correcting leaks"

Procedure: Correcting leaks

Materials needed:

- Electronic leak detector
- 1/4-inch wrench

1. Use the electronic leak detector to check all areas of the inlet that are potential sources of a leak. Potential leak areas are:
   - The plugged column connection
   - The septum nut, if present
   - The cooling tower assembly, if present
2. Correct leaks, using the wrench if necessary to tighten connections. You may need to repeat the leak test again to check for leaks.

3. If the pressure drop is now 0.03 psi/min or less, you can consider the inlet system leak-free.

   If the pressure drops faster than the acceptable rate, continue to search for leaks and repeat the pressure test. If all fittings appear to be leak free, but the inlet system is still losing too much pressure, you may need to replace the inlet manifold. Contact your Agilent service representative.

17 The Programmable Temperature Vaporization Inlet

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- Sampling heads

Columns and Traps

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- Additional temperature ramps

Cooling the inlet
- Configuring the PTV
- Shutdown behavior

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- Hot split introduction

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- Procedure: Using split mode with the column defined
- Procedure: Using split mode with the column not defined

Pulsed modes

Control table parameters—pulsed split mode
- Procedure: Using pulsed split mode with the column defined
- Procedure: Using pulsed split mode with the column not defined

Using the Splitless Modes

Flow patterns

Temperature considerations
- Cold splitless introduction
- Hot splitless introduction

Control table parameters—splitless operation
- Starting values
- Procedure: Using splitless mode with the column defined
- Procedure: Using splitless mode with the column not defined

Pulsed splitless mode operation
- Control table parameters—pulsed splitless operation
- Procedure: Using pulsed splitless mode with the column defined
- Procedure: Using pulsed splitless mode with the column not defined

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Procedure: Changing the septum

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Procedure: Replacing liners
Replacing the split vent trap filter cartridge
Procedure: Leak testing the gas plumbing
Procedure: Leak testing the PTV inlet
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Potential leak points

Consumables and replaceable parts

The Programmable Temperature Vaporization Inlet

Introducing the Agilent PTV

Operating modes

The Agilent Programmed Temperature Vaporization (PTV) Inlet System has five operating modes:

- The *split mode* is generally used for major component analyses.
- The *pulsed split mode* is like the split mode, but with a pressure pulse applied to the inlet during sample introduction to speed the transfer of material to the column.
- The *splitless mode* is used for trace analyses.
- The *pulsed splitless mode* allows for a pressure pulse during sample introduction.
- The *solvent vent mode* is used for large volume injection. Either single or multiple injections can be made for each run.

System requirements

The PTV inlet can be used with both manual and automatic injection.

For automatic multiple injections (large volume injections), an Agilent GC or MSD ChemStation is required. This function is not available under 6890 control alone. See "Using the Solvent Vent Mode" .

System components

1. The pneumatics module, located at the top rear of the GC.
2. The inlet body, always mounted in the front inlet position.
3. The trap, which is in the split line and placed to the left of the pneumatics carrier at the top rear of the chromatograph.

4. The coolant control valve. For liquid nitrogen, this valve is on the left outside wall of the oven. For liquid carbon dioxide, it is in the pneumatics carrier. These valves are *not* interchangeable—if you change coolants, you must also change all of the coolant plumbing including the valve and inlet jacket.
5. The thermocouple conversion board. It converts thermocouple readings from the inlet for use by the GC and is near the trap.

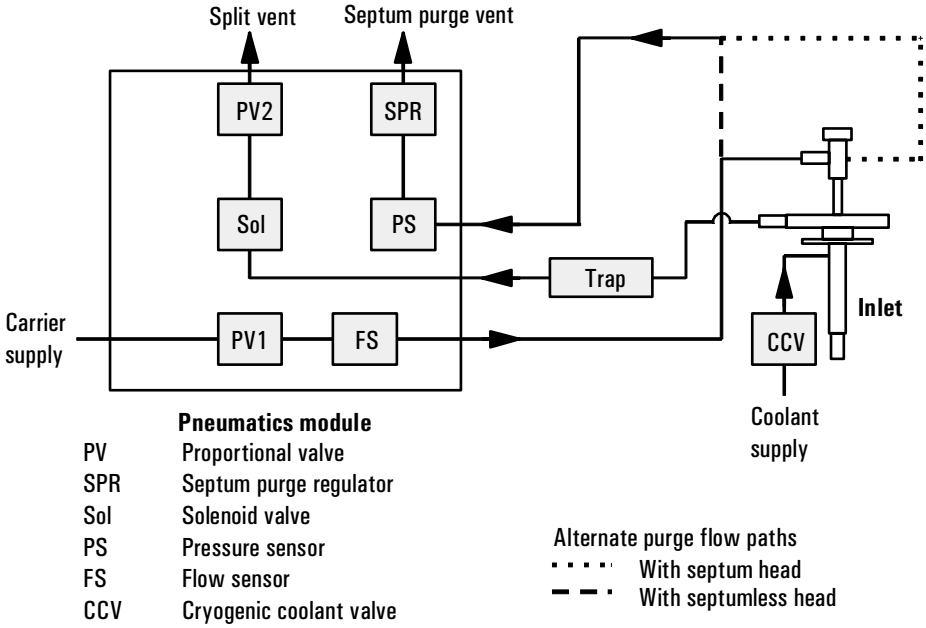


Figure 56 PTV system components

Sampling heads

Two heads are available for the PTV inlet.

- The septum head uses either a regular septum or a Merlin Microseal™ to seal the syringe passage. A stream of gas sweeps the inner side of the septum and exits through the septum purge vent on the pneumatics module. It may be used with either automatic or manual injection.

Caution At inlet temperatures below 40°C, the Merlin Microseal may not seal effectively—use a regular septum instead.

- The septumless head uses a check valve instead of a septum to seal the syringe entrance passage. It may be used with either automatic or manual injection.

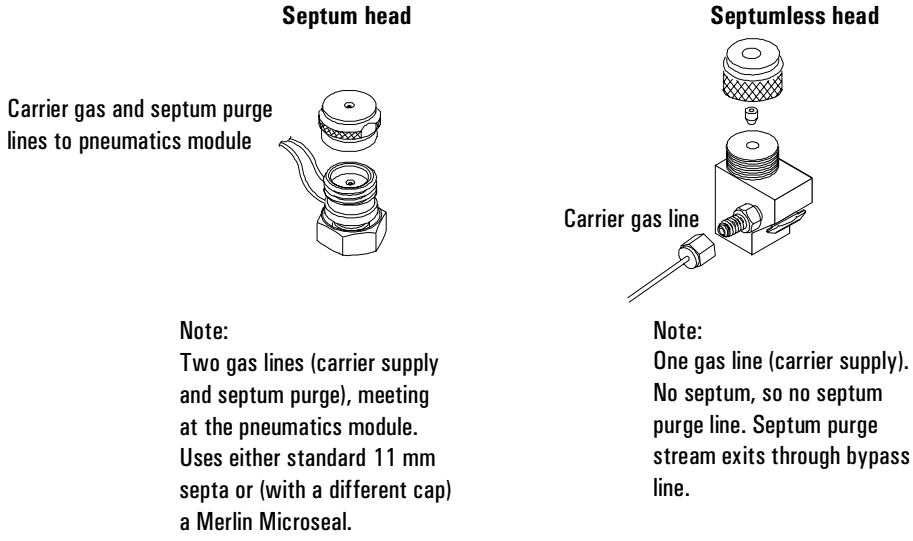


Figure 57 Sampling heads

The flow diagrams in the rest of this book show the septum head in place with a separate drawing for the septumless head plumbing.

Heating the inlet

The control parameters for PTV temperature programming are the same as for the column oven, but are reached by pressing [Front Inlet]. Temperature can be programmed with an initial temperature and up to 3 rates and plateaus. Rates between 0.1 and 720°C/min can be selected. See "Configuring the oven" for details.

Caution If the initial inlet temperature and the oven initial temperature are too close, the inlet may be unable to maintain its setpoint. We recommend a difference of at least 6°C, either higher or lower.

Additional temperature ramps

For most purposes, the PTV is designed to hold the sample in the inlet liner until the entire sample—there could be several injections—has been injected. Then the PTV is heated rapidly to transfer the sample to the column. This can be accomplished with an initial hold, a single ramp, and a hold at the end to complete sample transfer.

Two additional ramps are available and have several possible uses:

- The inlet can be heated to a high temperature to thermally clean the liner for the next run.
- The inlet can be programmed downward—just set the Final temp below the previous temperature—to reduce thermal stress on the inlet.
- Downward programming can be used to prepare the inlet for the next run. This can reduce cycle time for greater sample throughput.

Cooling the inlet

The sample may be injected into either a cooled or heated chamber. The initial chamber temperature can be reduced to –60°C (with CO_2 cooling) or to –160°C (with liquid N_2 cooling).

Caution If the initial inlet temperature and the oven initial temperature are too close, the inlet may be unable to maintain its setpoint. We recommend a difference of at least 6°C, either higher or lower.

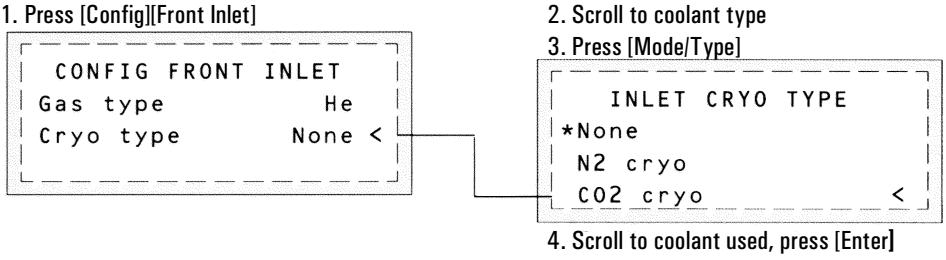
The 6890 GC supports only one type of coolant at a time.

Once a coolant is selected for any cryogenic device, that same coolant must be used for all such devices, including the column oven.

Since the GC can sense which coolant is used by the oven, if oven cooling is installed that coolant becomes the one that must be used by all other cooling devices.

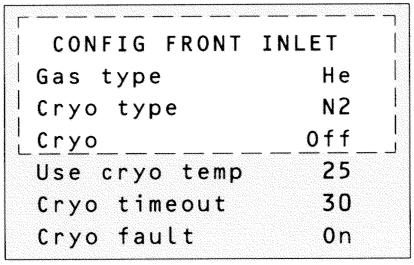
Configuring the PTV

To configure the PTV, press [Config] [Front Inlet]. If the inlet has not been configured previously, this screen is displayed.



If oven cooling is installed, your choices are restricted to the coolant used by the oven or `None`. If oven cooling is not installed, you must specify the coolant using the procedure in the figure.

If the Cryo type selection is anything other than `None`, several other parameters appear.



`Cryo` [ON] enables cryogenic cooling of the inlet as soon as the column oven reaches its initial temperature. [OFF] disables cooling.

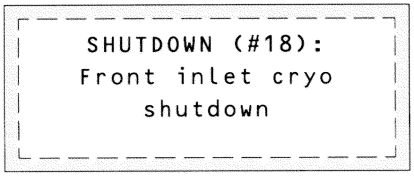
`Use cryo temp` If `Cryo` is ON, this is the upper limit of temperatures at which cryo cooling is used to hold the inlet at its setpoint. If the setpoint is higher than this limit, cryogenic cooling is used to bring the inlet down to its setpoint but is not used to hold it at the setpoint.

`Cryo timeout` Cryo timeout occurs, and the inlet temperature shuts down, when a run does not start within a specified time (range 5 to 120 minutes, default 30 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. A Post Sequence method could also be used.

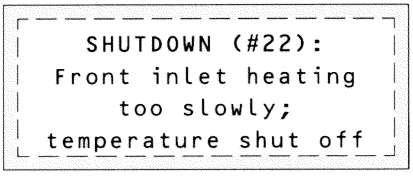
`Cryo fault` Shuts down the inlet temperature if it does not reach setpoint in 16 minutes of continuous cryo operation. Note that this is the time to *reach* the setpoint, not the time to stabilize and become ready at the setpoint.

Shutdown behavior

Both Cryo timeout and Cryo fault can cause cryo shutdown. If this happens, the inlet heater is turned off and the cryo valve closes. The GC beeps and displays this message:



The inlet heater is monitored to avoid overheating. If the heater remains on at full power for more than 2 minutes, the heater is shut down. The GC beeps and displays this message:



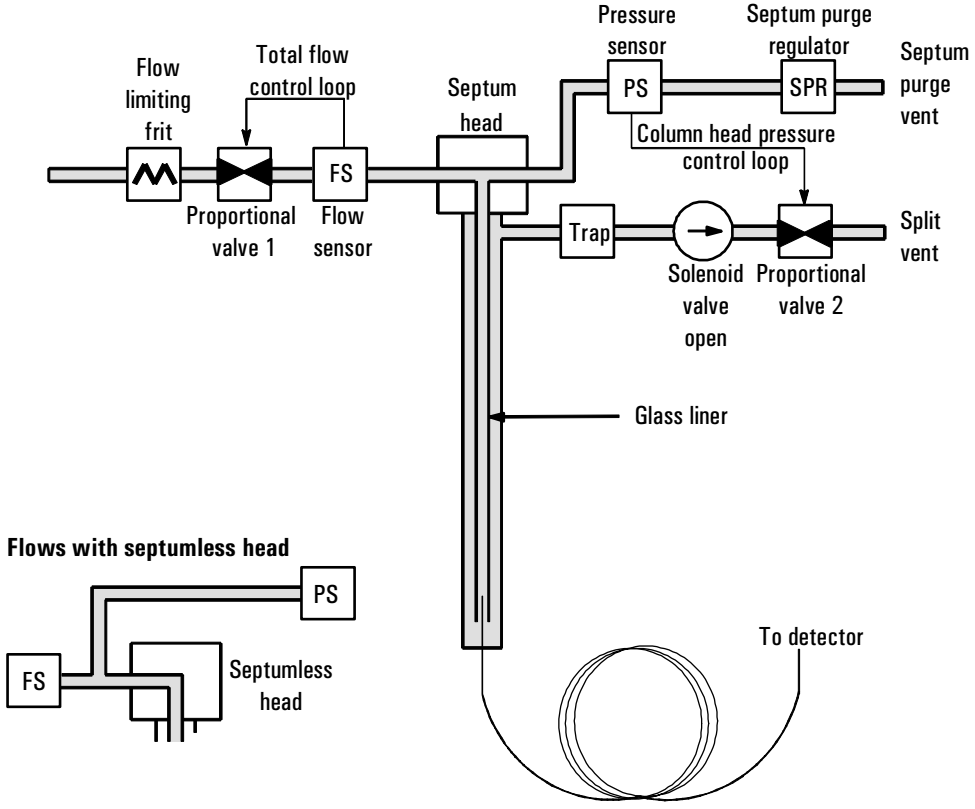
To recover from either condition, turn the GC off, then on, or enter a new setpoint.

Using the Split Modes

Flow pattern

The two split modes—with or without a pressure pulse—divide the gas stream entering the inlet between the column flow, the split vent flow through the solenoid valve, and the septum purge flow. The ratio of the split vent flow to the column flow is called the split ratio.

The main figure shows the flows with the septum head. Flows with the septumless head are the same except that the septum purge flow bypasses the head (lower left).



Temperature considerations

Cold split introduction

For cold split sample introduction, use an initial inlet temperature below the normal boiling point of the solvent. If the liner volume is enough to hold all the vaporized solvent, start the first inlet temperature ramp at 0.1 minutes with a high heating rate (500°C/min or higher). The final temperature should be high enough to volatilize the heaviest analytes from the liner and should be held for at least 5 minutes. A final temperature of 350°C for 5 minutes has proven sufficient to quantitatively transfer C_{44}.

For larger injection volumes or to eliminate the solvent, hold the initial temperature long enough to vent the solvent through the Split vent and then begin the first ramp. Use a fast rate for thermally stable analytes. Slower rates may help minimize thermal degradation in the inlet.

A single temperature ramp is enough for the injection process. The remaining ramps may be used to clean the liner or to reduce the inlet temperature in preparation for the next injection.

Hot split introduction

For hot split introduction, set an initial temperature high enough to volatilize the analytes. No additional thermal parameters are required as the inlet will maintain the setpoint throughout the run.

Because of the small liner volume (about 120 microliters), the PTV has a limited injection capacity with hot split introduction. Injection volumes exceeding 1 µL in the hot split mode may overflow the inlet causing analytical problems. Cold split introduction avoids this potential problem.

Control table parameters—split mode operation

`Mode:` The current operating mode—split

`Temp` Actual and setpoint inlet initial temperatures.

`Init time` Hold time at the inlet initial temperature.

`Rate #` Temperature program rate for inlet thermal ramps 1, 2, and 3.

`Final temp #` Final inlet temperature for ramps 1, 2, and 3.

`Final time #` Hold time at Final temp 1, 2, and 3.

`Pressure` Actual and setpoint inlet pressure.

`Split ratio` The ratio of split flow to column flow. Column flow is set at the Column 1 or Column 2 control table. This line does not appear if your column is not defined.

`Split flow` Flow, in mL/min, from the split/purge vent. This line does not appear if your column is not defined.

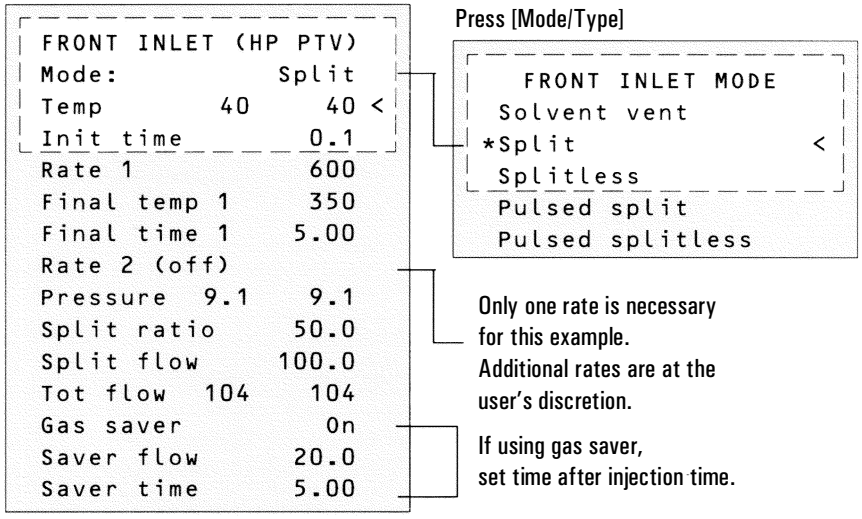
`Total flow` These are the actual and setpoint values of the total flow into the inlet, which is the sum of the split flow, column flow, and septum purge flow. When you change the total flow, the split ratio and split flow change while the column flow and pressure remain the same.

Procedure: Using split mode with the column defined

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet].
   a. Scroll to `Mode`: and press [Mode/Type]. Select `Split`.
   b. Set the inlet temperature and any desired ramps.
   c. If you want a specific split ratio, scroll to `Split ratio` and enter that number. The split flow will be calculated and set for you.
   d. If you want a specific split flow, scroll to `Split flow` and enter that number. The split ratio will be calculated and displayed for you.

$$\text{Split ratio} = \frac{\text{Split flow}}{\text{Column flow}}$$

e. If desired, turn on `Gas saver`. Set the `Saver time` after the injection time.



3. Press [Prep Run] before manually injecting the sample if the Gas Saver is on (see page 285).

Procedure: Using split mode with the column not defined

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control" .
2. Press [Front Inlet].
   a. Set temperature.
   b. Set total flow into the inlet. Measure flows out of the split vent and septum purge vent using a flow meter.
   c. Subtract the septum purge flow from `Total flow` to get split flow.

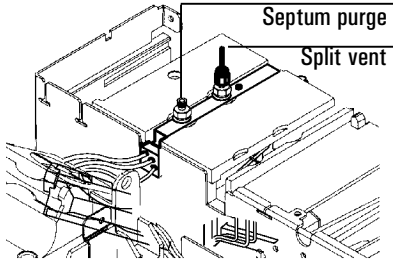
d. Calculate the split ratio. Adjust as needed.

```
FRONT INLET (HP PTV)
Mode:              Split
Temp         40      40 <
Init time             0.10
Rate 1                 600
Final temp 1           350
Final time 1          5.00
Rate 2 (off)
Pressure 10.0         10.0
Tot flow 80.3         80.3
```

Press [Mode/Type]

```
   FRONT INLET MODE
 Solvent vent
*Split                  <
 Splitless
 Pulsed split
 Pulsed splitless
```

Only one rate is necessary for this example. Additional rates are at the user's discretion.



Front of instrument

Pulsed modes

The pressure pulse modes (split and splitless) increase inlet pressure just before the beginning of a run and return it to the normal value after a specified amount of time. The pressure pulse sweeps the sample out of the inlet and into the column faster, reducing the chance for sample decomposition in the inlet. If your chromatography is degraded by the pressure pulse, a retention gap may help restore peak shape.

You must press the [Prep Run] key before doing manual injections in the pressure pulse mode.

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

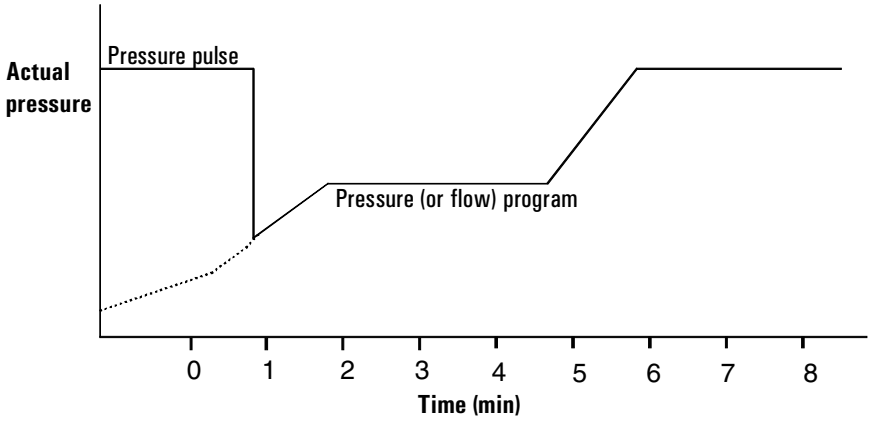


Figure 58 Pressure pulse and column flow or pressure

Control table parameters—pulsed split mode

`Mode:` The current operating mode—pulsed split.

`Temp` Actual and setpoint inlet temperatures.

`Init time` Hold time at the initial inlet temperature.

`Rate #` Temperature program rate for inlet thermal ramps 1, 2, and 3.

`Final temp #` Final inlet temperature for ramps 1, 2, and 3.

`Final time #` Hold time at Final temp 1, 2, and 3.

`Pressure` Actual and setpoint inlet pressure before and after the pressure pulse. This is the starting point of a pressure program or the fixed pressure if a program is not used.

`Pulsed pres` The inlet pressure you desire at the beginning of a run. The pressure rises to this setpoint after [Prep Run] is pressed and remains constant until `Pulse time` elapses, when it returns to `Pressure`.

`Pulse time` Inlet pressure returns to its normal setpoint at this time after Start Run.

`Split ratio` The ratio of split flow to column flow. Column flow is set at the Column 1 or Column 2 control table. This line does not appear if your column is not defined.

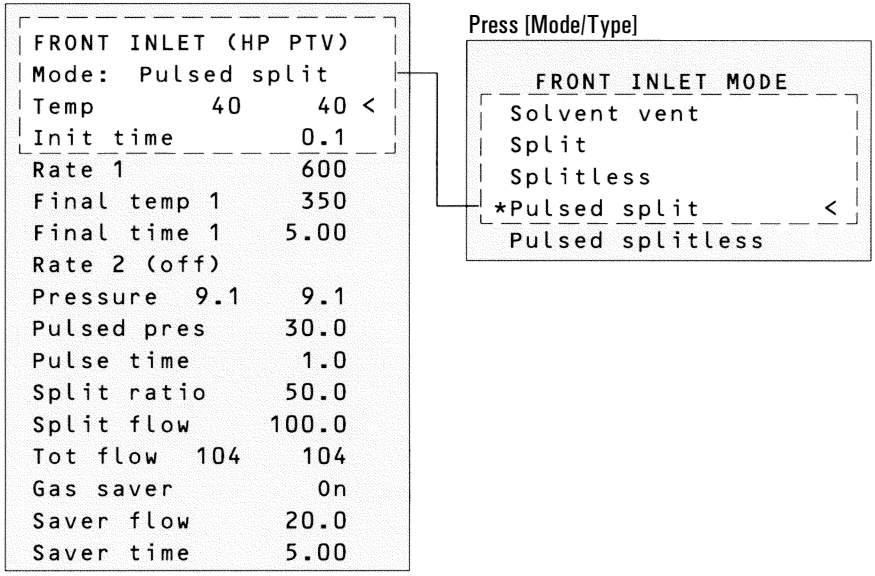
`Split flow` Flow, in mL/min from the split/purge vent. This line does not appear if your column is not defined.

`Total flow` The total flow into the inlet, the sum of the split flow, column flow, and septum purge flow. When you change total flow, the split ratio and split flow change while column flow and pressure remain the same. When a pressure pulse is used, total flow increases to keep the split ratio constant.

Procedure: Using pulsed split mode with the column defined

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet].
   a. Scroll to `Mode`: and press [Mode/Type]. Select `Pulsed Split`.
   b. Set the inlet temperature and any desired ramps.
   c. Enter values for `Pulsed Pres` and `Pulse time`.
   d. If you want a specific split ratio, scroll to `Split ratio` and enter that number. The split flow is calculated and set for you.
   e. If you want a specific split flow, scroll to `Split flow` and enter that number. The split ratio is calculated and displayed for you.
   f. Turn `Gas saver` on, if desired. Set the time greater than `Pulse time`.

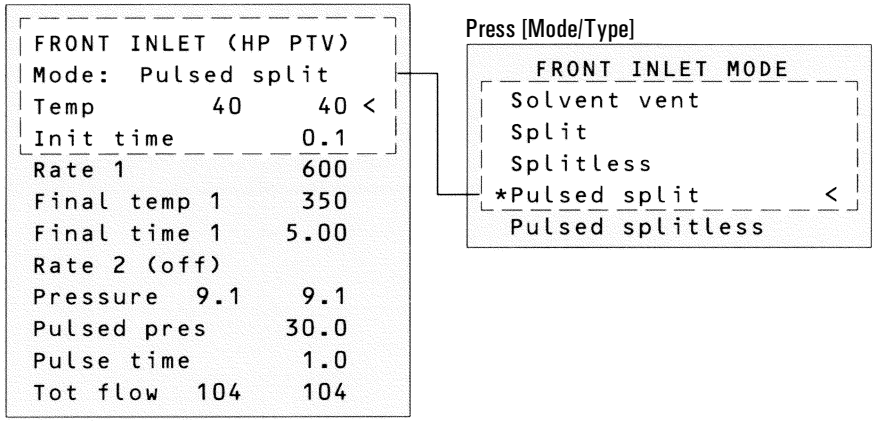
Split ratio = $\frac{\text{Split flow}}{\text{Column flow}}$



3. Press [Prep Run] (see page 285) before injecting a sample manually.

Procedure: Using pulsed split mode with the column not defined

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet].
   a. Scroll to `Mode:` and press [Mode/Type]. Select `Pulsed Split`.
   b. Set the inlet temperature and any desired ramps.
   c. Enter values for `Pulsed Pres` and `Pulse time`.
   d. Set total flow into the inlet. Measure flows out of the split vent and septum purge vent using a flow meter.
   e. Subtract the septum purge flow from `Total flow`.
   f. Calculate the split ratio. Adjust as needed.



Using the Splitless Modes

Flow patterns

In these modes—with or without a pressure pulse—the solenoid valve is closed during injection and vaporization of the sample and stays so while the sample transfers to the column. At a specified time after injection, the valve opens to sweep vapors left in the liner out the split vent. This avoids solvent tailing due to the large inlet volume and small column flow rate. The main figure shows the flows with the septum head. Flows with the septumless head are the same except that the septum purge flow bypasses the head (lower left).

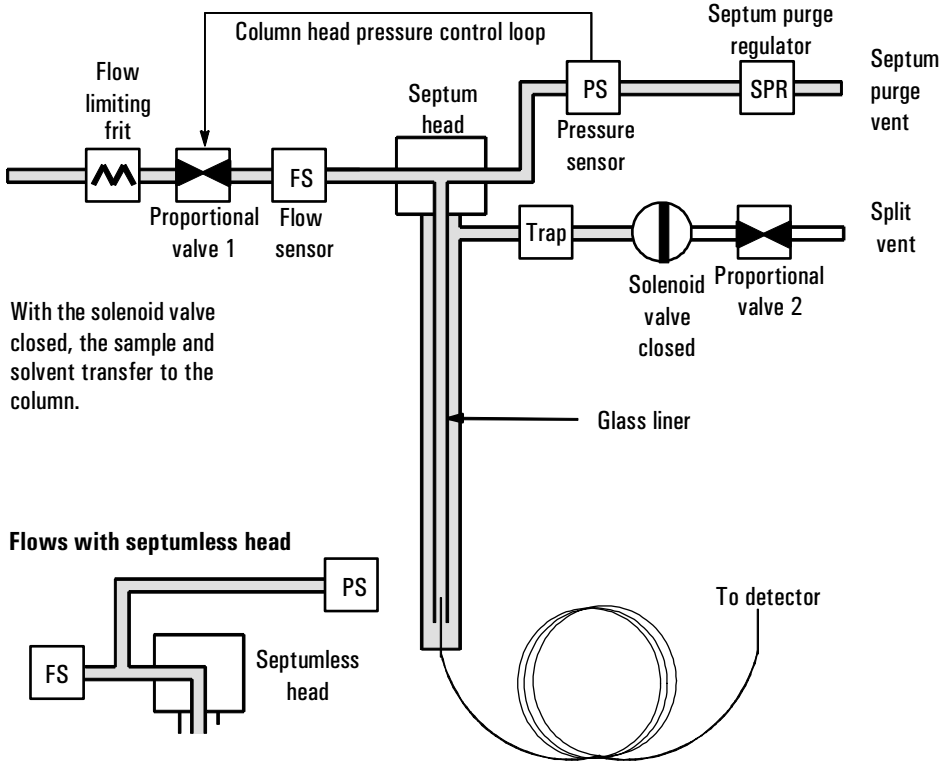


Figure 59 Stage 1. Sample injection

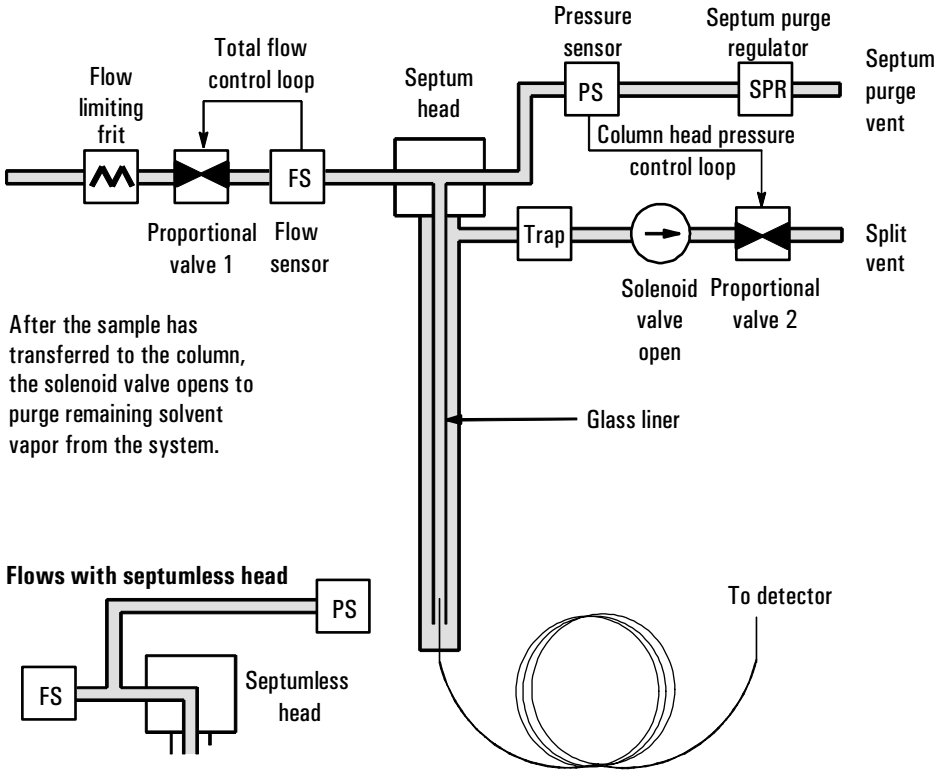


Figure 60 Stage 2. Purging

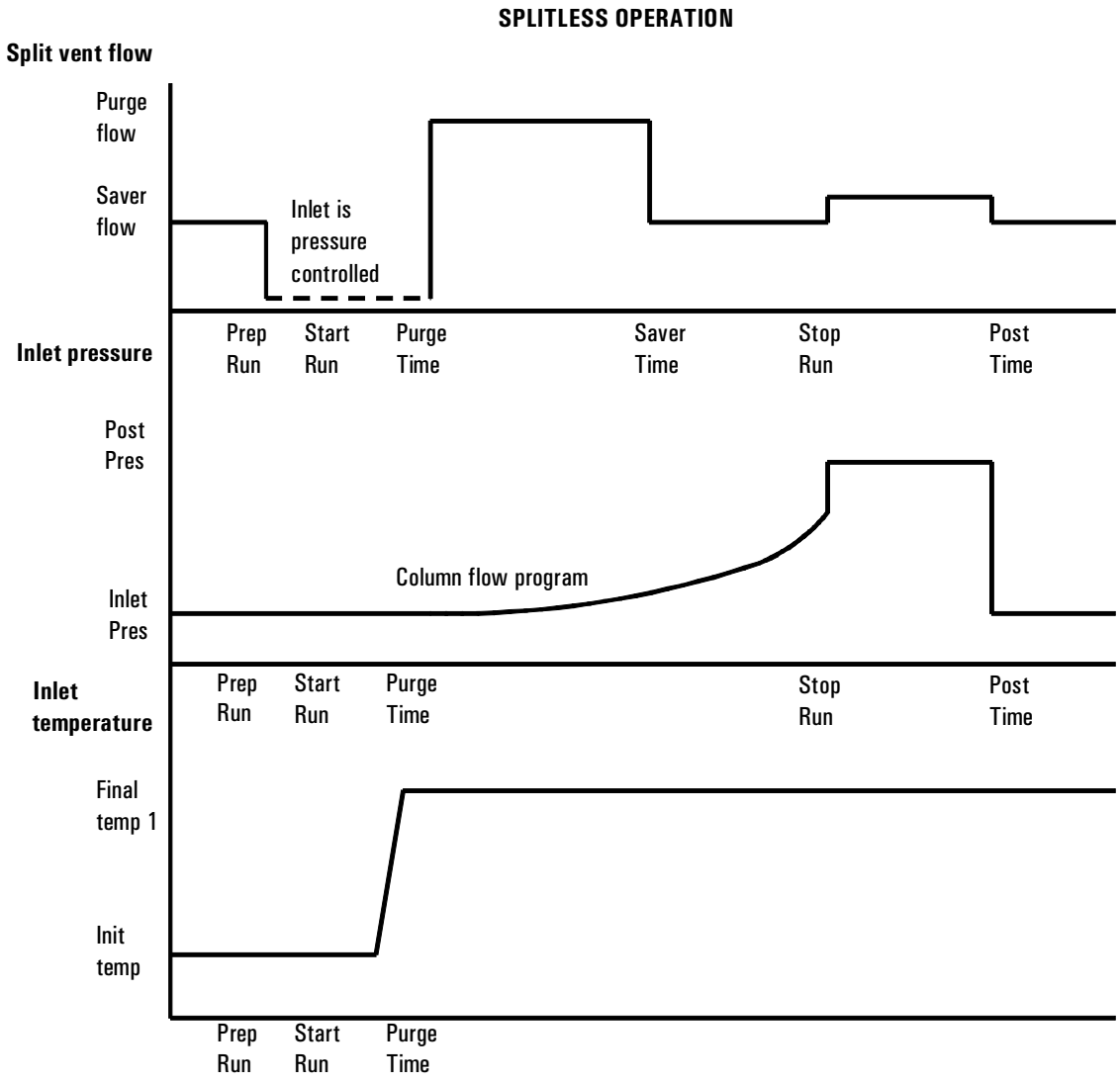


Figure 61 Flows, pressures, and temperatures

Temperature considerations

Cold splitless introduction

For cold splitless introduction, use an initial inlet temperature below the normal boiling point of the solvent. For most solvents, starting the first inlet temperature ramp at 0.1 minutes provides good transfer and reproducibility. A program rate of 500°C/min or higher is appropriate for thermally stable analytes. A final temperature of 350°C, held for 5 minutes, has quantitatively transferred up to C_{44} alkane.

A main advantage of temperature programmability is that the inlet can be heated gently to transfer delicate analytes. If the oven temperature is initially low enough to refocus the analytes on the column, the inlet heating rate can be made slower (e.g., 120°C/min). This reduces thermal degradation from the inlet and can improve peak shape and quantitation.

For most applications of cold splitless, a single temperature ramp is enough. The remaining ramps can be used to clean the liner or to decrease the inlet temperature to prepare for the next injection.

Hot splitless introduction

For hot splitless introduction, select an initial temperature high enough to volatilize the analytes. No additional temperature parameters are required as the inlet will maintain the setpoint throughout the run.

Because of the small liner volume (about 120 µL), the PTV cannot contain vapor resulting from large liquid injection volumes. Injection volumes greater than 1 µL may overflow vapor from the inlet, causing analysis variations. Cold splitless introduction avoids this problem.

Control table parameters—splitless operation

`Mode:` The current operating mode—splitless.

`Temp` Actual and setpoint inlet temperatures.

`Init time` Hold time at the initial inlet temperature.

`Rate #` Temperature program rate for inlet thermal ramps 1, 2, and 3.

`Final temp #` Final inlet temperature for ramps 1, 2, and 3.

`Final time #` Hold time at Final temp 1, 2, and 3.

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

`Purge time` The time, after the beginning of the run, when you want the purge valve to open.

`Purge flow` The flow, in mL/min, from the purge vent, at `Purge time`. You will not be able to specify this value if operating with your *column not defined*.

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

Starting values

A successful splitless injection consists of these steps:

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

Some experimentation is needed to refine the operating conditions. Table 43 provides starting values for the critical parameters.

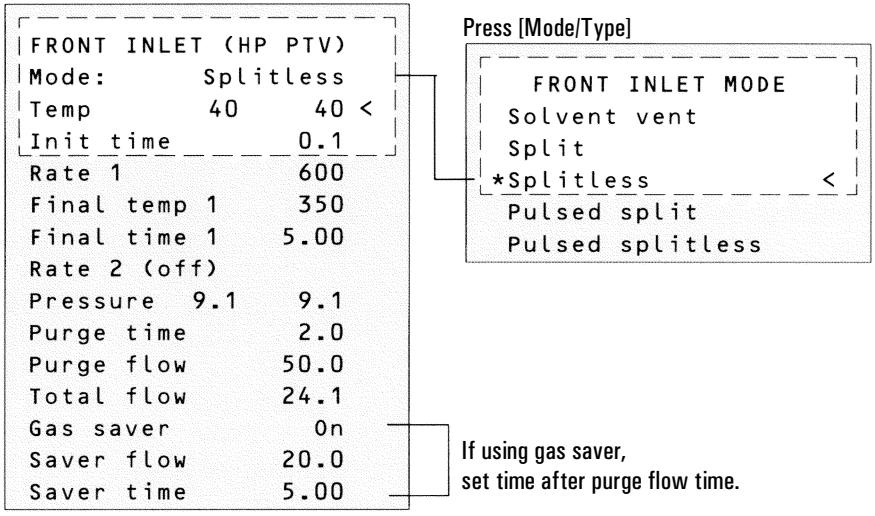
Table 43 Splitless Mode Inlet Parameters

Parameter	Allowed setpoint range	Suggested starting value
Oven temperature	No cryo, ambient + 10° C to 450° C CO_2 cryo, –60° C to 450° C N_2 cryo, –80° C to 450° C	10°C below solvent boiling point
Oven initial time	0 to 999.9 minutes	≥ Inlet purge time
Inlet purge time	0 to 999.9 minutes	$\frac{\text{Liner volume*}}{\text{Column flow}}$ x 5
Gas saver time	0 to 999.9 minutes	After purge time
Gas saver flow	15 to 1000 mL/min	15 mL/min greater than maximum column flow
Inlet temperature	No cryo, oven temp + 10°C CO_2 cryo, –50°C to 450°C N_2 cryo, –60°C to 450°C	10°C below solvent boiling point for 0.1 min, then ramp up

* Liner volume is about 120 µL

Procedure: Using splitless mode with the column defined

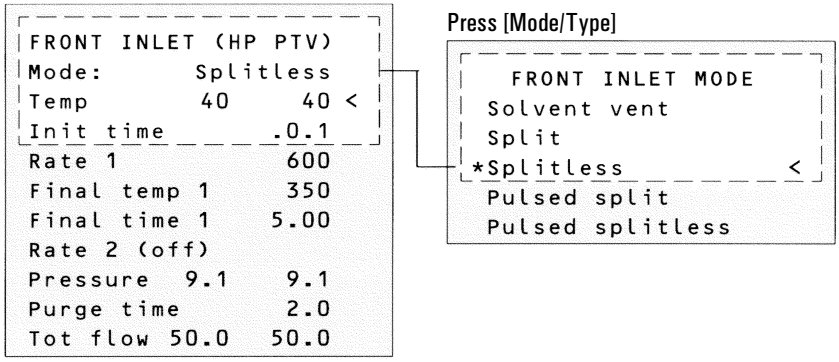
1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet].
   a. Scroll to `Mode`: and press [Mode/Type]. Select `Splitless`.
   b. Set the inlet temperature and any desired ramps.
   c. Enter a purge time and a purge flow.
   d. If desired, turn `Gas saver` on. Make certain the time is set *after* the purge flow time.



3. Press [Prep Run] (see page 285) before manually injecting a sample.

Procedure: Using splitless mode with the column not defined

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet].
   a. Scroll to `Mode`: and press [Mode/Type]. Select `Splitless`.
   b. Set the inlet temperature and any desired ramps.
   c. Enter a purge time.
   d. Set your total flow greater than the column flow plus the septum purge flow (about 3 to 6 mL/min) to guarantee adequate column flow.



3. Press [Prep Run] (see page 285) before manually injecting a sample.

Pulsed splitless mode operation

See page 389 for a discussion of the pulsed pressure modes.

Control table parameters—pulsed splitless operation

`Mode`: The current operating mode—pulsed splitless.

`Temp` Actual and setpoint inlet temperatures.

`Init time` Hold time at the initial inlet temperature.

`Rate #` Temperature program rate for inlet thermal ramps 1, 2, and 3.

`Final temp #` Final inlet temperature for ramps 1, 2, and 3.

`Final time #` Hold time at Final temp 1, 2, and 3.

`Pressure` Actual and setpoint inlet pressure before and after the pressure pulse. It sets the starting point of a pressure program or the fixed pressure if a program is not used.

`Pulsed pres` The inlet pressure you desire at the beginning of a run. The pressure rises to this setpoint after [Prep Run] is pressed and remains constant until `Pulse time` elapses, when it returns to `Pressure`.

`Pulse time` Pressure returns to its normal setpoint at this time.

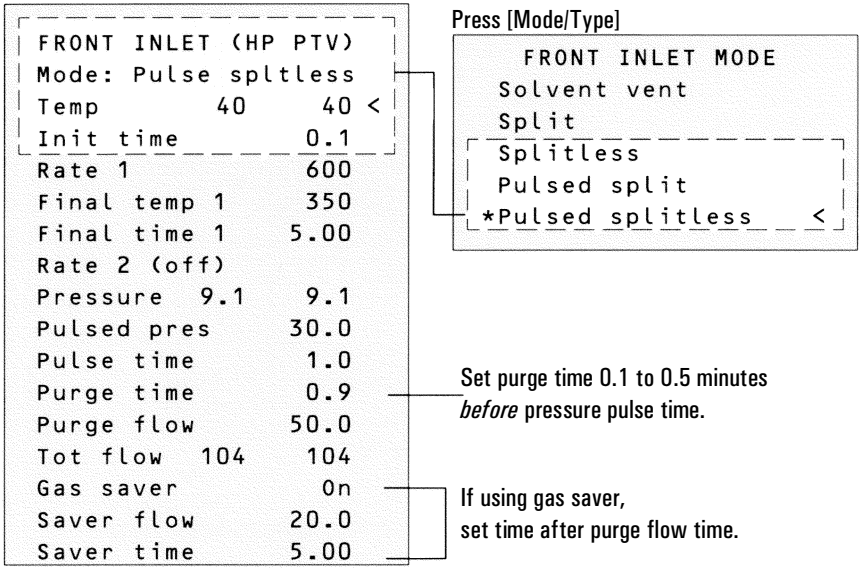
`Purge time` The time, after the beginning of the run, that you wish the purge valve to open. Set purge time 0.1 to 0.5 minutes before pulse time.

`Purge flow` The flow, in mL/min, from the purge vent, at `Purge time`. The column must be defined.

`Total flow` This is the total flow into the inlet, representing a total of the column flow and the septum purge flow.

Procedure: Using pulsed splitless mode with the column defined

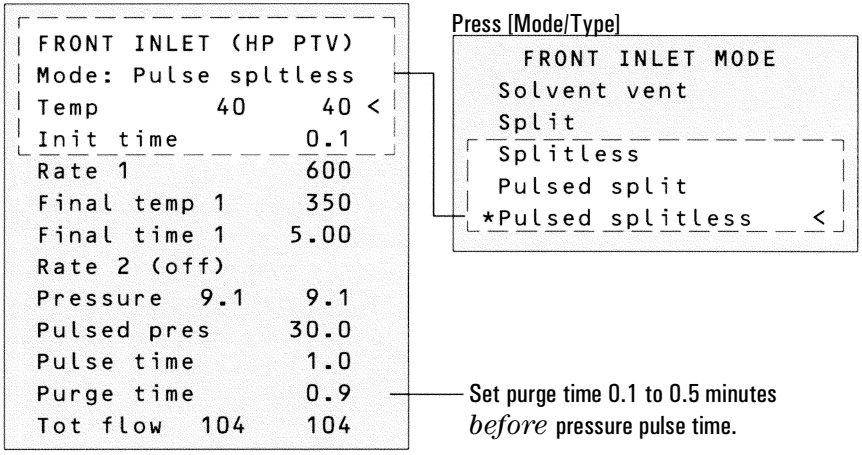
1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet].
   a. Scroll to `Mode:` and press [Mode/Type]. Select `Pulsed Splitless`.
   b. Set the inlet temperature and any desired ramps.
   c. Enter values for `Pulsed pres` and `Pulse time`.
   d. Enter the `Purge time` when you wish the purge valve to open.
   e. Enter a `Purge flow`.
   f. Turn `Gas saver` on, if desired. Set the time *after* the purge flow time.



3. Press [Prep Run] (see page 285) before manually injecting a sample.

Procedure: Using pulsed splitless mode with the column not defined

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet].
   a. Scroll to `Mode:` and press [Mode/Type]. Select `Pulsed Splitless`.
   b. Set the inlet temperature and any desired ramps.
   c. Enter values for `Pulsed Pres` and `Pulse time`.
   d. Enter the `Purge time` when you wish the purge valve to open.
   e. Enter a `Purge flow`.



3. Press [Prep Run] (see page 285) before manually injecting a sample.

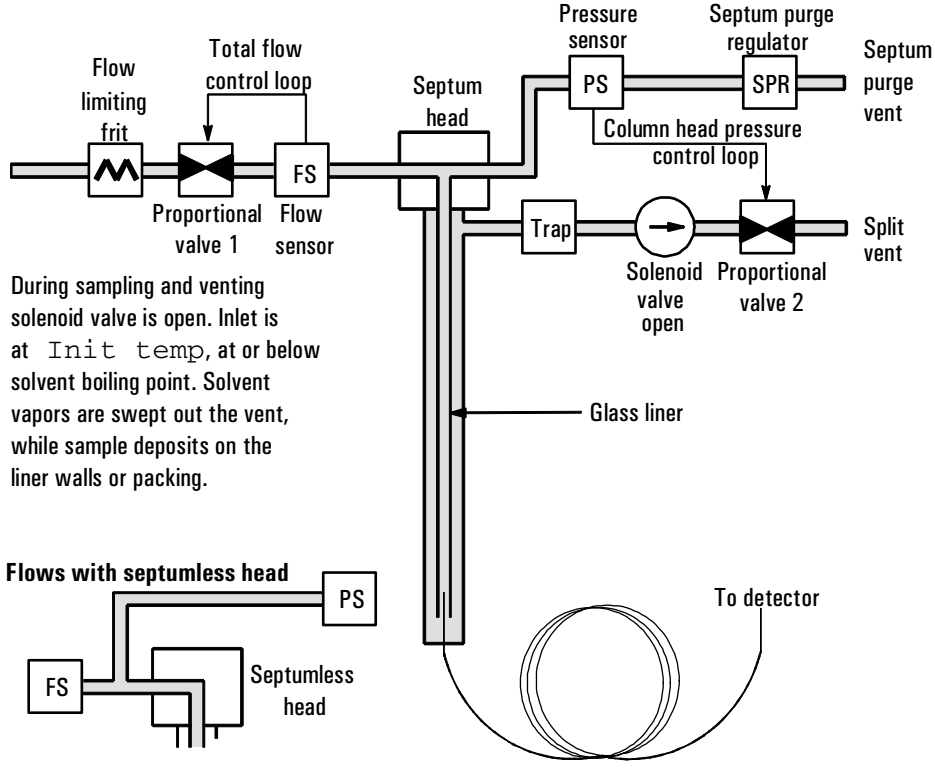
Using the Solvent Vent Mode

Flow patterns

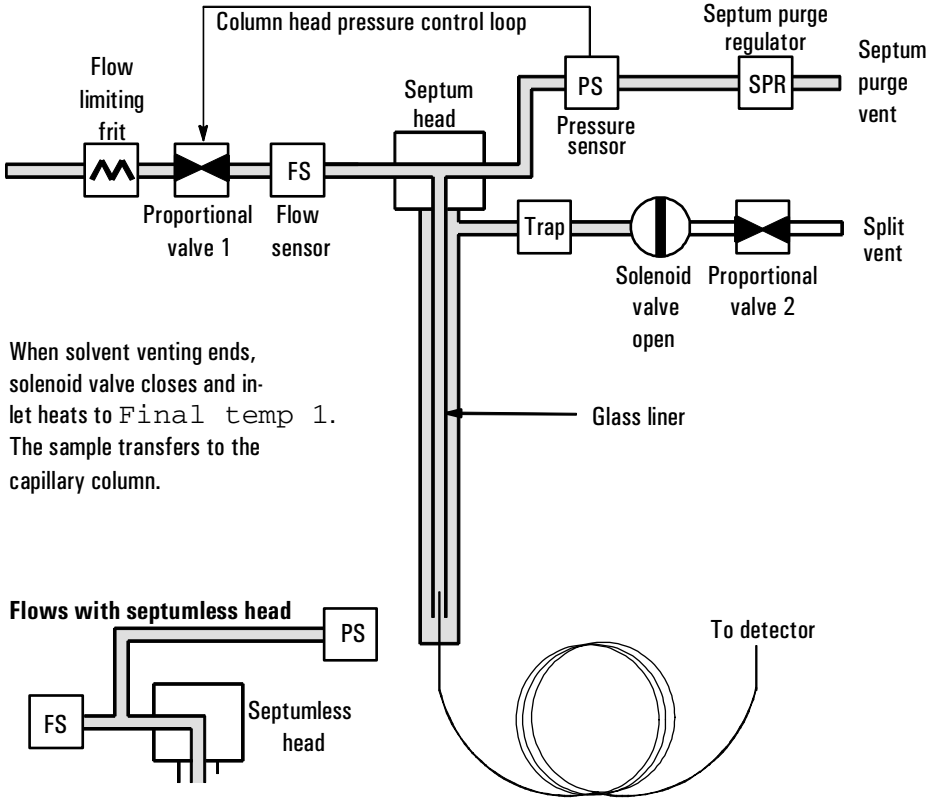
The sample is injected into a cold inlet. If conditions are properly chosen and the sample is suitable, analytes deposit in the inlet liner while the solvent evaporates and is swept out. Large or multiple injections can be used to concentrate sample in the inlet before transferring to the column for analysis.

The main figure shows the flows with the septum head. Flows with the septumless head are the same except that the septum purge flow bypasses the head (lower left).

Stage 1. Sample and vent



Stage 2. Sample transfer



Stage 3. Purge and cleanup

The solenoid valve opens again and the system returns to the Stage 1 configuration but with different setpoints. The PTV inlet is flushed. Additional ramp rates are available to thermally clean the inlet or to reduce inlet temperature after sample transfer. This can extend the life of the liner.

Temperature, pressure, and flow considerations

The solvent vent mode goes through three distinct pneumatic states; venting, sample transfer, and purging. The vent portion allows the inlet pressure and the vent flow to be adjusted to optimize solvent elimination. The transfer state mimics traditional splitless operation and transports the analytes from the liner to the column. The purging mode allows the user to prepare the inlet for the next run.

A fundamental difficulty with solvent vent mode is the potential loss of volatile analytes with the solvent. Several solutions are possible for this situation:

- The inlet liner can be packed with a more retentive material, such as Tenax. This greatly improves volatile analyte recovery but may impact recovery of higher boiling materials.
- Some of the solvent can be left in the liner when sample transfer begins. The residual solvent acts like a stationary phase and retains volatile material, but at the expense of a larger solvent peak.
- The inlet temperature can be reduced. This reduces the vapor pressure of the volatile analytes and permits higher recoveries.

Solvent removal can be speeded up by:

- Reducing pressure in the inlet during sample introduction—the `Vent pressure` parameter
- Increasing flow through the inlet—the `Vent flow` parameter

While all these possibilities do complicate use of the PTV, they provide increased flexibility and new potential to solve difficult problems.

Sequence of operations

These are the steps in a typical analysis using the solvent vent mode.

Step		Parameter	Value
1	**Before injection**	Flow at split vent	Either Purge flow or Saver flow
		Inlet pressure	Derived from column setpoint
	The system is resting, with Purge flow (or Saver flow, if on) through the inlet.		
2	**Prep Run begins**	Flow at split vent	Vent flow setpoint
		Inlet pressure	Vent pressure setpoint
	Setpoints change to prepare for injection. When GC is ready, the sample is injected. Inlet and oven temperature program Init times begin. Solvent venting and analyte trapping begin.		
3	**At Vent end time**	Flow at split vent	None, solenoid valve closed
		Inlet pressure	Column pressure setpoint
	Solvent venting ends, analyte transfer begins as inlet heats up.		
4	**At Purge time**	Flow at split vent	Purge flow setpoint
		Inlet pressure	Column pressure setpoint
	Analyte transfer ends, inlet is purged of residual vapor. Analysis begins.		
5	**At Saver time**	Flow at split vent	Saver flow setpoint
		Inlet pressure	Column pressure setpoint
	Analysis ends, carrier flow reduced to save gas (if Saver is on).		

Some important points

- The flow through the column is governed by the pressure in the inlet. This is controlled, during the analysis part of the process, by the flow or pressure setpoint or program entered *for the column*.
- The controlling times must be in the order shown; Vent end time *before* Purge time *before* Saver time.
- Vent end time must occur before the inlet starts to heat and release analytes.
- Purge time must occur before the oven begins to heat and move sample through the column.

Timelines

Time increases downward; all other quantities increase to the right.

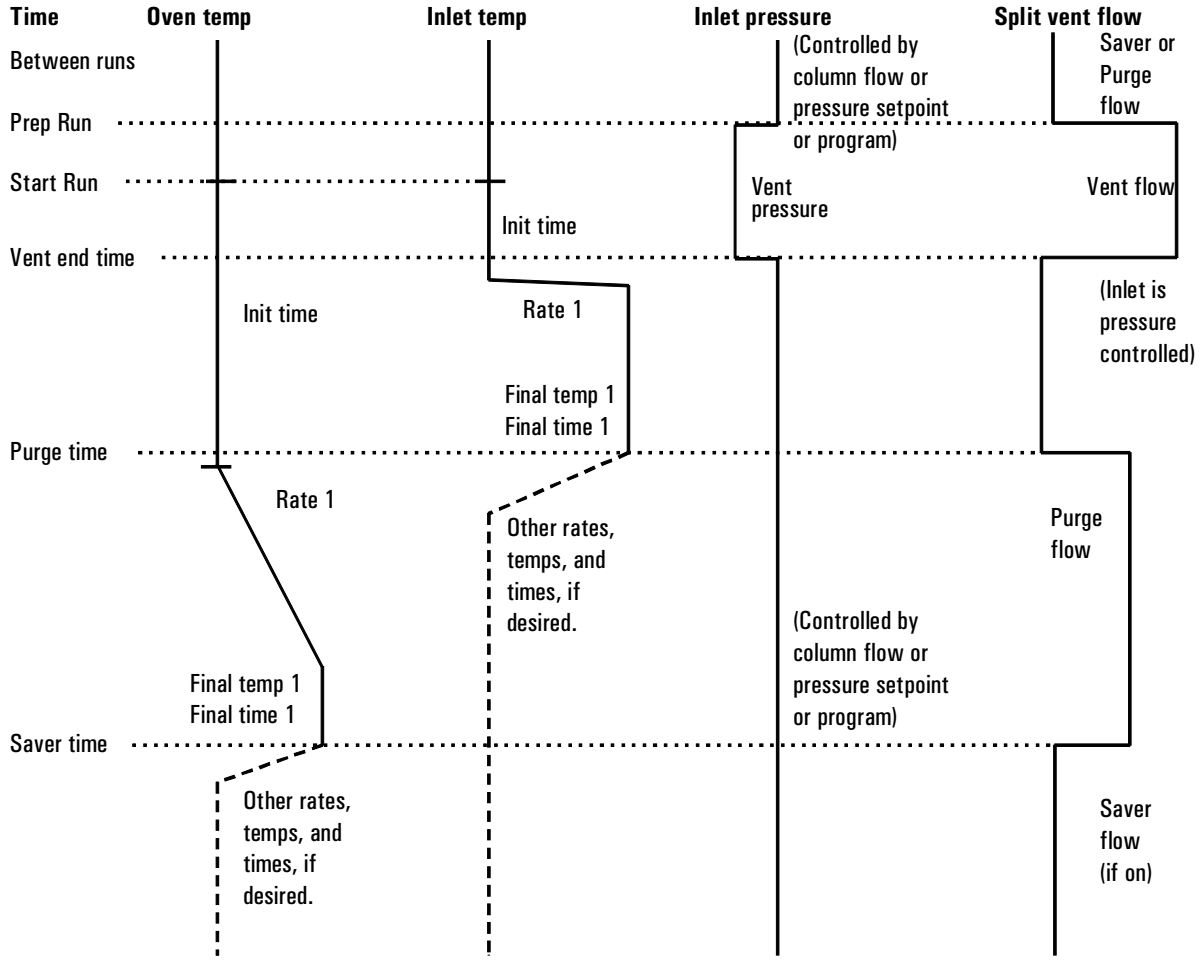


Figure 62 Time relationships

When is Start Run?

Both the inlet and oven temperature programs begin at Start Run. All times—such as Purge time—are measured from Start Run. When does Start Run occur?

- If the sample is injected manually, Start Run occurs when the user presses the Start Run key.
- If a single injection per run is made using an autosampler, Start Run occurs when the syringe carrier moves down to make the injection.
- If multiple injections per run are made using an autosampler, Start Run occurs when the syringe carrier moves down to make the first injection of the set. There are no Start Run signals for the rest of the injections in the set. These additional injections take time. The inlet and oven temperature programs, mainly the `Init time` values, must be adjusted to allow for this. So must the various time values that control the inlet operation. This is discussed in more detail under "Large volume injection".

Control table parameters—solvent vent operation

`Mode:` The current operating mode—solvent vent.

`Temp` Actual and setpoint initial inlet temperatures.

`Init time` The time, measured from Start Run, when the initial inlet temperature hold ends. Usually greater than `Vent end time`.

`Rate #` Temperature program rate for inlet thermal ramps 1, 2, and 3.

`Final temp #` Final inlet temperature for ramps 1, 2, and 3.

`Final time #` Hold time at Final temp 1, 2, and 3. This time is a duration; it is *not* measured from Start Run.

`Pressure` Actual and setpoint inlet pressure before and after the vent period. It sets the starting point of column head pressure.

`Vent pressure` The inlet pressure during the vent period. By decreasing the inlet pressure while venting, solvent elimination proceeds faster. Also, the pressure reduction decreases the amount of carrier gas—and solvent vapor—that enters the column during this time.

Users select from 0 to 100 psig. If 0 is chosen, the inlet uses the lowest pressure possible at the given vent flow. Table 44 shows approximate values for this minimum at various vent flows of helium. Pressures less than those in the table are not possible unless the flow is reduced.

Table 44 Minimum attainable pressures

Vent flow (mL/min)	Actual vent pressure at "0" psig setpoint	Actual vent pressure at "0" kPa setpoint
50	0.7	5
100	1.3	10
200	2.6	18
500	6.4	44
1000	12.7	88

`Vent flow` The flow of carrier gas out the split vent during the vent period. Higher flows sweep the liner more quickly and reduce the time for solvent elimination. For most columns, 100 mL/min vent flow eliminates solvent at an acceptable rate but puts minimal material on the column.

`Vent end time` The time, measured from Start Run, when solvent venting ends. For large volume injections, this time is normally greater than the time for the injection to complete.

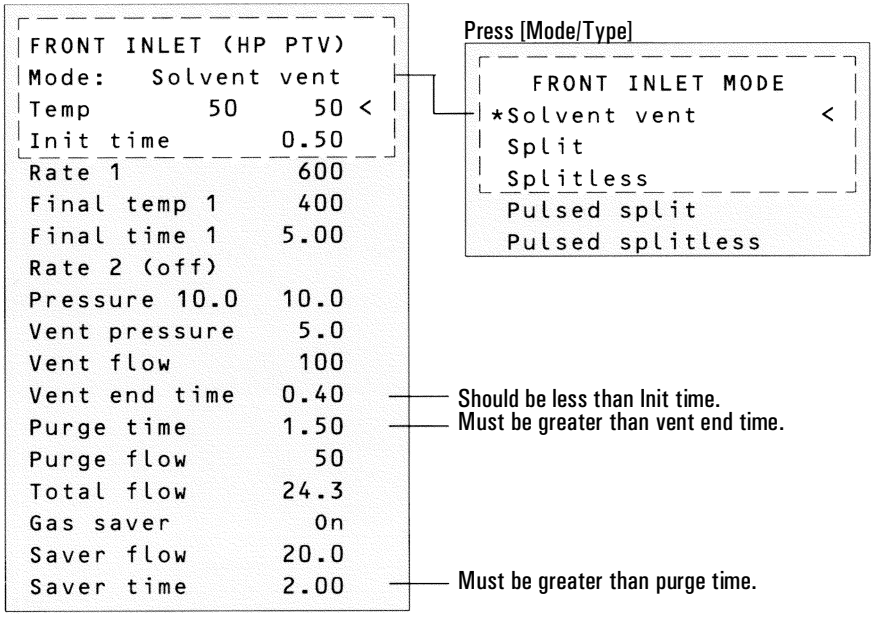
`Purge time` The time, measured from Start Run, when sample transfer ends. It began at `Vent end time`.

`Purge flow` The flow of carrier gas to the inlet beginning at `Purge time`.

`Total flow` The Total flow displays the actual flow to the inlet.

Procedure: Using solvent vent mode with the column defined

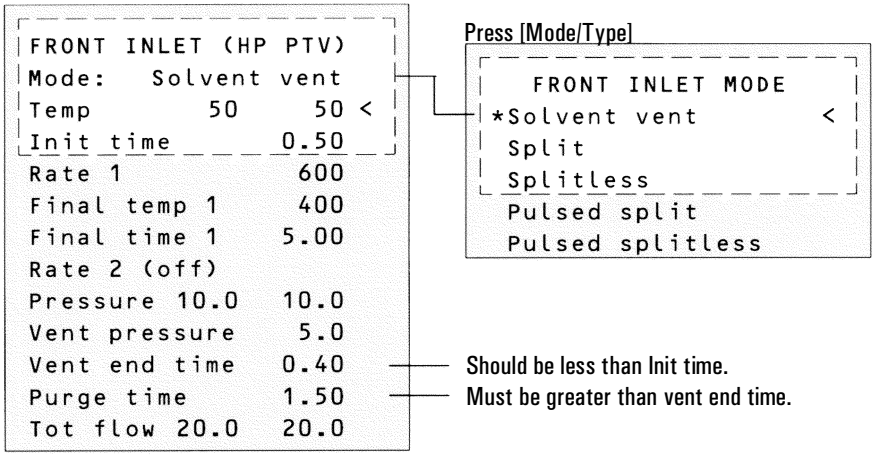
1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet].
   a. Scroll to `Mode`: and press [Mode/Type]. Select `Solvent vent`.
   b. Enter a vent pressure, a vent flow, and a vent end time.
   c. Set the inlet temperature and ramps, as desired.
   d. Enter a purge time and a purge flow.
   e. If desired, turn `Gas saver` on. Make certain the time is set *after* the purge time.



3. Press [Prep Run] (see page 285) before manually injecting a sample.

Procedure: Using solvent vent mode with the column not defined

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet].
   a. Scroll to `Mode`: and press [Mode/Type]. Select `Solvent vent`.
   b. Enter a vent end time and a vent pressure.
   c. Set the inlet temperature and ramps, as desired.
   d. Enter a purge time. It must be greater than the vent end time.
   e. Set total flow greater than the column flow plus the septum purge flow (about 6 mL/min) to guarantee adequate column flow.



3. Press [Prep Run] (see page 285) before manually injecting a sample.

Large volume injection

Most vaporizing inlets are designed for liquid injections in the 1 to 5 µL range. With larger injections, the vapor cloud created when the sample vaporizes may overflow the inlet and degrade the chromatography. For the PTV, the nominal liner liquid capacities are:

Table 45 Liner capacities

Liner	Nominal liquid capacity	Inertness
Open baffle	5 µL	High
Glass wool packed	25 µL	Lower, because of greater surface area

In the solvent vent mode, analytes are thermally trapped in the liner while the solvent is removed. With the solvent gone, the liner volume can be used for another injection. Injection can be repeated several times to concentrate the analytes from a large sample volume. After injection and solvent removal, the analytes are transferred to the column. This can replace the need for offline concentrating and minimize loss of sample.

Multiple injections by an automatic sampler can be used with the PTV to achieve large volume injection. A ChemStation controls the process.

ChemStation requirements

A GC or MSD ChemStation is necessary for multiple injection because the needed parameters are not available through the 6890 GC keyboard.

- GC ChemStation Software revision A.04.02 or later
  or Software revision A.04.01 *plus* the software provided with the PTV.
- MSD ChemStation Software revision A.03.00 or later

Table 46 Control parameters—Injector configuration subscreen

Parameter	Range	Default
Syringe size	0.1 to 100 µL	10 µL
Nanoliter adapter	Present or not present	Not present
Multiple injections	Single or multiple	Single

- `Syringe size` Full volume of the syringe.
- `Nanoliter Adapter` Controlled by a checkbox. If checked, indicates that a nanoliter adapter is present on the injector. If *not* checked, means that a nanoliter adapter is not present on the injector. The adapter is **always** present on the G2613A injector
- `Multiple Injections` Controlled by a checkbox. If checked, the sampler makes multiple injections into the inlet for each run according to the other parameters. It issues a Start Run command at the first injection only. If *not* checked, the sampler makes one injection—and issues a Start Run command—for each run. This is the default mode of operation.

Table 47 Control parameters—Injector screen

Parameter	Range	Default
Inject **X** µL **Y** times	**X**: 0.1 to 0.5 × syringe volume **Y**: 1 to 100	**X**: 0.1 × syringe volume **Y**: 1
Delay between injections	0 to 100 seconds	0
Preinjection washes	0 to 15	0
Postinjection washes	0 to 15	0
Pumps	0 to 15	0

- `Inject X µL Y times` **X** is the amount to be injected; **Y** is the number of injections to make. If the nanoliter adapter is checked on the Injector Configuration screen, the range becomes 0.02 to 0.4 x syringe volume.
- `Delay` A pause time, in seconds, between injections. This is added to the minimum hardware cycle time.
- `Preinjection washes` Number of times to wash the syringe with solvent and/or sample *before the first injection*. No washes are performed before the rest of the injections in a multiple injection set.

- `Postinjection washes` Number of times to wash the syringe with solvent *after the last injection*. No washes are performed after the rest of the injections in a multiple injection set.
- `Preinjection pumps` Number of times to pump the syringe plunger before drawing up the measured sample. Pump are performed only before the first injection of a multiple injection set.

Calculated values

The software calculates and displays:

- On the Injector screen: `Total` Product of X (Volume per injection) and Y (Injections per run).
- On the Inlets screen: `Estimated total injection time` The approximate total time, in minutes, to make a set of multiple injections based on the parameters entered and the mechanical cycle time of the sampler. Includes `Delay between injections`, pre- and post-injection dwell times, and viscosity delays.

An example

These values were used for a sample with a broad range of boiling points.

General parameters	
Name	**Value**
Sample	C_{10} to C_{44} hydrocarbons in hexane
Mode	Solvent vent
PTV liner	Glass wool packed
Injection volume	One 10.0 µL injection (25 µL syringe)
Injection speed	Fast
Column	30 m x 320 µm x 0.25 µm -5, p/n 19091J-413
Column flow	4 mL/min constant flow

Inlet parameters			
Name	**Value**	**Name**	**Value**
Init temp	40°C	Rate 2 (off)	
Init time	0.3 min	Pressure	15.6 psig
Rate 1	720°C/min	Vent pressure	0.0 psig
Final temp 1	450°C	Vent flow	100 mL/min
Final time 1	5 min	Vent end time	0.2 min
Rate 2	100°C/min	Purge time	2.0 min
Final temp 2	250°C	Purge flow	50 mL/min
Final time 2	0 min		

Oven parameters	
Name	**Value**
Init temp	40°C
Init time	2.5 min
Rate 1	25°C/min
Final temp 1	320°C
Final time 1	10.0 min
Rate 2 (off)	

Detector parameters	
Name	**Value**
Detector	FID
Detector temp	400°C
Hydrogen flow	40 mL/min
Air flow	450 mL/min
Makeup (N_2)	45 mL/min

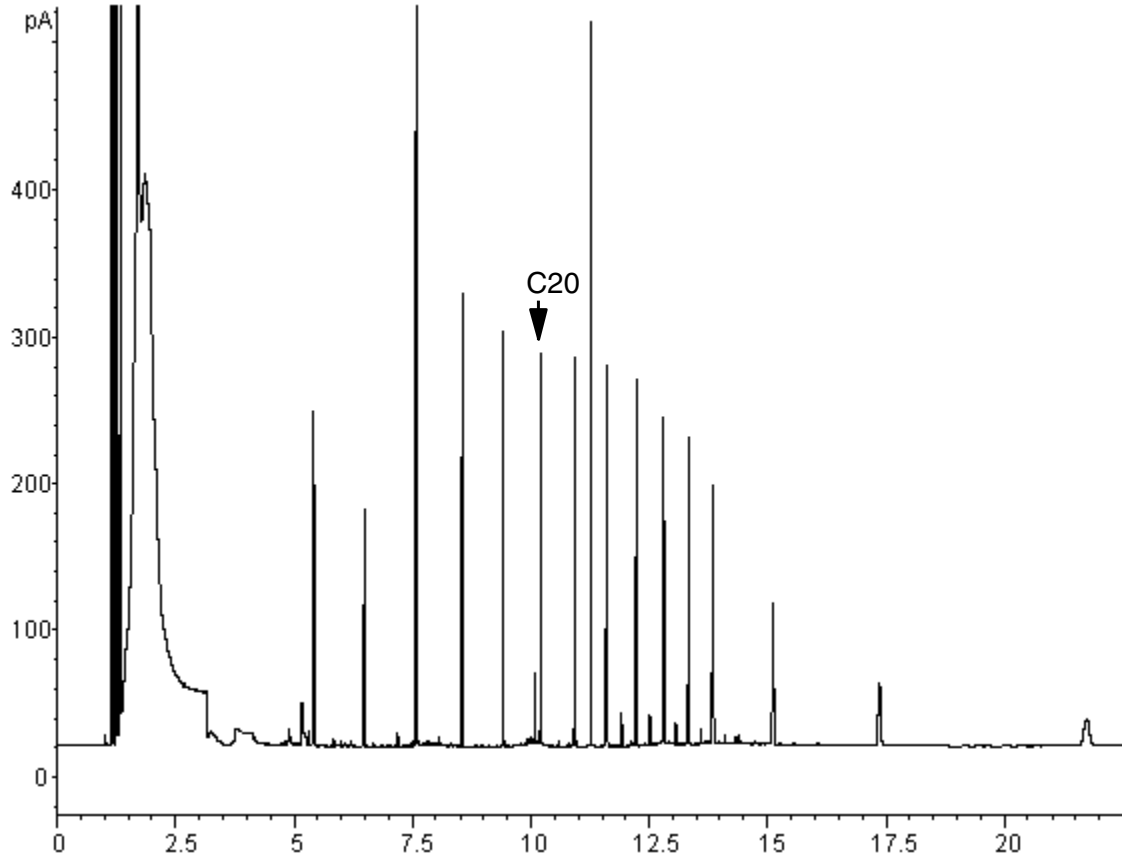


Figure 63 Chromatogram from one 10 µL injection

These results were compared with a splitless analysis of the same sample, which should produce 100% recovery of all analytes. The data showed that, under these conditions, compounds above C_{20} were completely recovered and that the recovery was independent of injection size; Compounds lower than C_{20} were partially vented with the solvent.

Possible adjustments

Depending on what you are trying to accomplish, you have a number of possible adjustments available.

To eliminate more solvent

- Increase the vent end time, inlet initial time, and purge time. This will not affect analytes that are quantitatively trapped but will eliminate more of the solvent peak.
- Increase the vent flow to sweep the liner more rapidly with the same inlet timing. Increasing vent flow raises vent pressure if it is set to 0. This puts more solvent onto the column.
- Raise the inlet initial temperature to vaporize more solvent and allow more to be eliminated. This also increases the loss of volatile analytes since their vapor pressures also increase.

To improve recovery of low boiling analytes

- Reduce inlet temperature to lower the vapor pressure of the analytes and trap them more effectively. This also reduces solvent vapor pressure and more time will be needed to eliminate it.
- Use a retentive packing in the liner. Materials such as Tenax permit higher recovery of volatile analytes but may not release higher boiling compounds. This must be considered if quantitation on these high boiling peaks is desired.
- Leave more solvent in the liner. The solvent acts as a pseudo stationary phase and helps retain volatile analytes. This must be balanced against the detector's tolerance for solvent.

An example—continued

The single injection example shown on the last few pages makes it clear that a 10 µL injection does not overload the glass wool packed liner. This means that multiple 10 µL injections are possible.

It was decided to make 10 injections per run, each of 10 µL size. This would increase analytical sensitivity substantially. No adjustments were made to

improve recovery of the low boilers since the purpose of this analysis was to detect and measure the high boiling components.

The ChemStation estimated that 10 injections would require a total of 1.3 minutes. The following timing changes were made:

Parameter	Increased from	To
Inlet Init time	0.3 minutes	1.6 minutes
Vent end time	0.2 minutes	1.5 minutes
Purge time	2.0 minutes	3.0 minutes
Oven Init time	2.5 minutes	3.0 minutes

The result is shown in Figure 27.

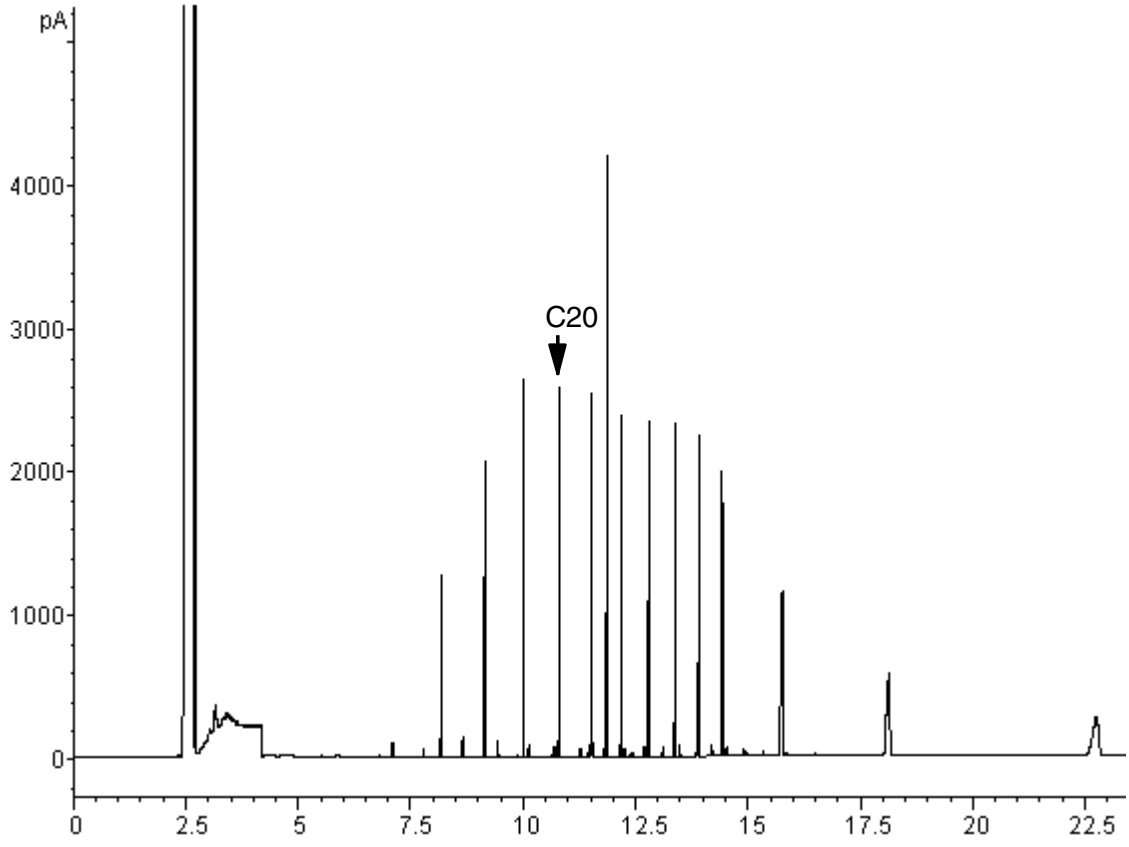


Figure 64 Chromatogram from ten 10 µL injections

Maintaining a PTV

Inlet adapters

The Graphpak™-2M connector (the inlet adapter) at the bottom of the inlet is sized to the column diameter. When a different diameter column is to be installed, the adapter must be changed.

The adapter number is stamped on the side of the adapters. Select the smallest hole diameter that will accept the column.

Table 48 Inlet adapters

Column ID	Inlet adapter number	Quantity	Part no.
200 µm	31	1	5182-9754
250 µm	45	1	5182-9761
320 µm	45	1	5182-9761
530 µm	70	1	5182-9762

Procedure: Replacing inlet adapters

1. Unscrew the column nut from the adapter. Remove the nut and the column from the inlet.
2. With a 6 mm wrench, remove the inlet adapter, being careful not to lose the silver seal inside. Save the adapter for later use.
3. Select the appropriate inlet adapter for the column to be installed. Insert a new silver seal (part number 5182-9763, pkg of 5) into the adapter and screw the adapter onto the inlet finger tight. Use the 6 mm wrench to tighten the adapter an additional 1/16- to 1/8-turn.

   Do not overtighten the adapter. The inlet can be damaged if the adapter is forced. If the adapter leaks, check the silver seal and replace it if necessary.

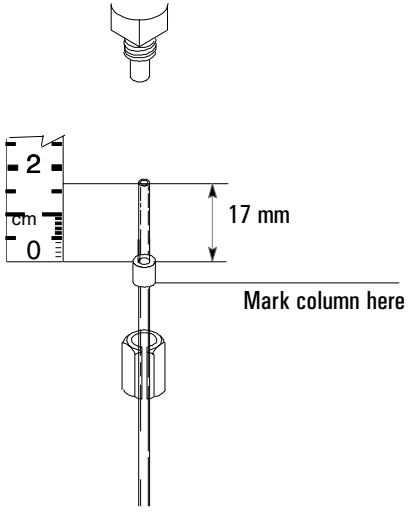
Procedure: Installing columns

Graphpak-2M ferrules are sized to the column outer diameter.

Table 49 Columns and ferrules

Column ID	Graphpak ferrule hole ID	Quantity	Part no.
200 µm	0.31 mm	10	5182-9756
250 µm	0.40 mm	10	5182-9768
320 µm	0.45 mm	10	5182-9769
530 µm	0.70 mm	10	5182-9770

1. Place the appropriate Graphpak ferrule onto the column inlet end and pull it at least 30 mm from the end.
2. With a glass knife or other fused silica cutter, remove approximately 10 mm from the column end to eliminate graphite contamination.
3. Position the ferrule so that it is 17 mm from the column end. Place a small mark (typewriter correction fluid is useful) at the back of the ferrule and, making sure that the column is correctly positioned, insert the column end into the adapter.



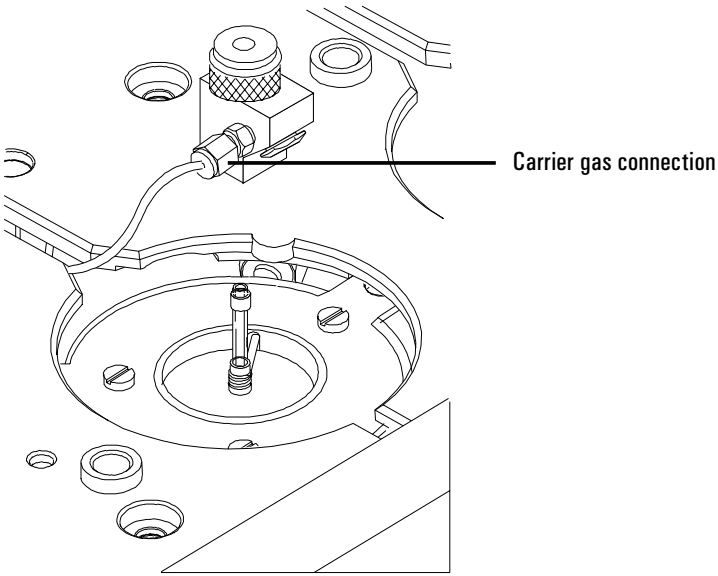
4. Screw the column nut on finger tight. Using a 5 mm wrench, tighten the column nut 1/8- to 1/4-turn. Be careful not to overtighten.
5. Check the connections for leaks. If there are any leaks at the column adapter, tighten it slightly more with the open end wrench provided.

The septumless head

This sampling head uses a check valve instead of a septum to seal the syringe entrance passage. It may be used with either automatic or manual injection. Syringes must have 23 gauge needles (see "Consumables and replaceable parts").

Procedure: Removing the septumless head

1. Cool the inlet to room temperature.
2. Disconnect the carrier gas line.
3. Unscrew the septumless head counterclockwise from the inlet.
4. Screw the new head onto the inlet. Tighten it 1/8-turn past finger tight.



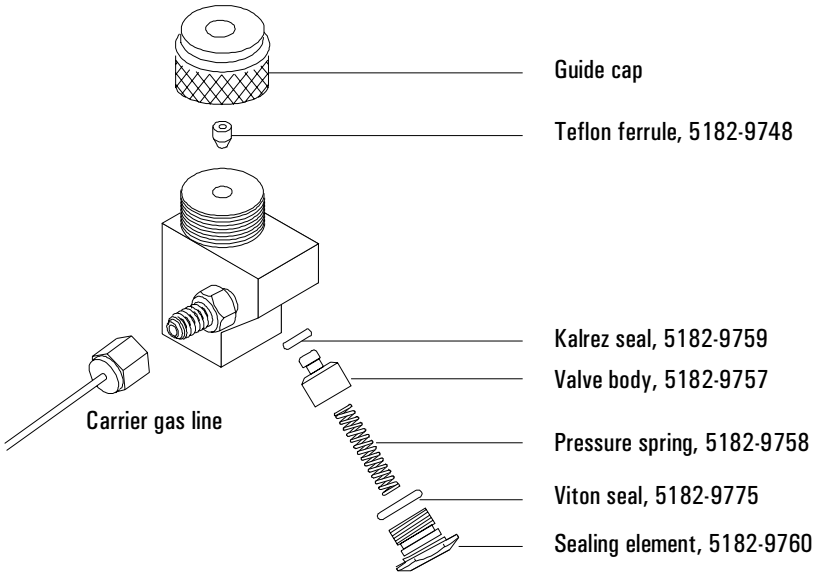
5. Reconnect the carrier gas line.

6. Check all connections on the sampling head for leaks. If necessary, tighten them again by hand.

Procedure: Cleaning the septumless head

Minor deposits from sample mixtures can collect in the head. Dust and abraded material particles can enter together with the syringe needle, eventually causing leaks. We recommend periodic cleaning.

1. Cool the inlet to room temperature.
2. Disconnect the carrier gas line and unscrew the head from the inlet.
3. Unscrew the sealing element from the head. Carefully remove the Viton seal and the pressure spring.



4. Unscrew the guide cap from the head and remove the Teflon ferrule.

Caution Do *not* use a sharp object to extract the valve body—this can leave scratches that cause leaks.

5. Insert a syringe with a 23 gauge needle carefully into the head to press the valve body with the Kalrez seal slightly out of the head. Carefully tap the head

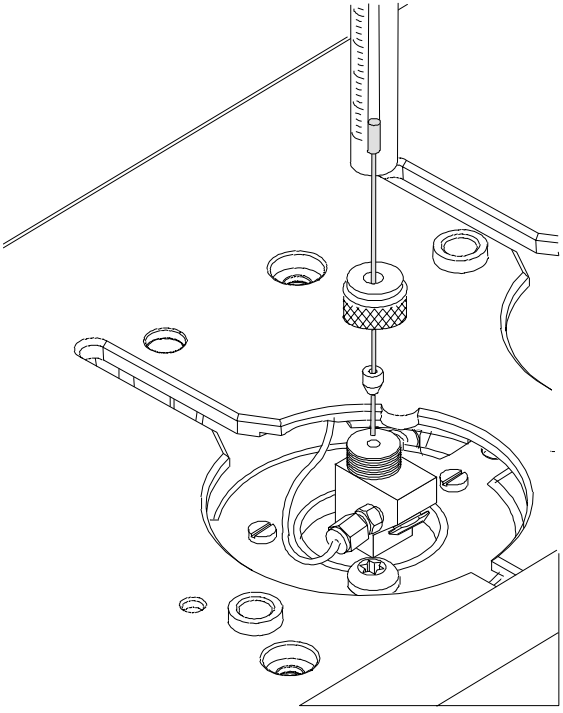
on a soft smooth surface so that the valve body falls out completely or slips so far out that you can grasp it with your fingers.

6. Remove the seal from the valve body.
7. Carefully clean all components in hexane.
8. Assemble the head in reverse order. Make sure that you work absolutely lint-free and that the seals and the pressure spring are not damaged.
9. Use this opportunity to check the Teflon ferrule. If it must be replaced, see page 426 for instructions.
10. Check the entire system again for leaks; if necessary, carefully retighten the guide cap slightly more with the syringe needle inserted and/or replace the Kalrez seal.

    If the head leaks when a syringe is inserted, the Teflon ferrule is the problem. If the head leaks without a syringe inserted, the seals may need to be replaced.

Procedure: Replacing the Teflon ferrule

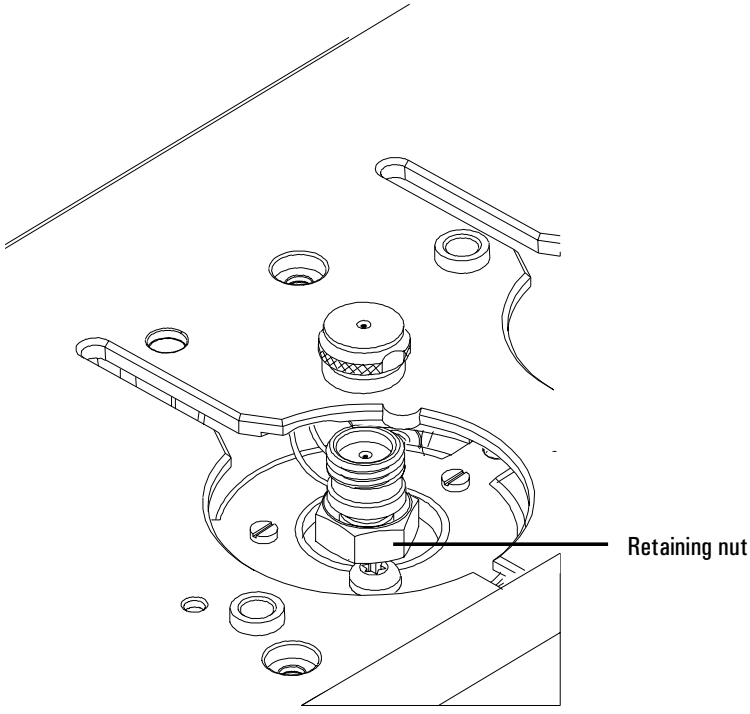
1. Unscrew the guide cap from the septumless head and remove the Teflon ferrule.



2. Push the guide cap and the new Teflon ferrule over the syringe needle so that at least 10 mm of the needle tip is exposed.
3. Guide the end of the syringe needle into the septumless head until the ferrule meets the septumless head.
4. Tighten the guide cap until resistance is first felt.
5. Check for leaks when the syringe needle has been fully introduced.
6. If necessary, carefully tighten the guide cap until the inlet stops leaking.

The septum head

The septum head uses either a regular septum or a Merlin Microseal to seal the syringe passage. A stream of gas sweeps the inner side of the septum and exits through the septum purge vent on the pneumatics module.



Procedure: Removing the septum head

The septum head connects to the inlet via a free-spinning retaining nut.

1. Cool the inlet to room temperature.
2. Use a 5/8-inch wrench to loosen the retaining nut on the septum head.
3. Gently remove the septum head assembly from the inlet. Be careful not to overly bend the 1/16-inch lines. For best results, lift the head to clear the inlet and then push it to either side to allow access.

4. To reinstall the septum head, gently align the head with the inlet and manually engage the free-spinning nut to the inlet.

   The nut should easily turn on to the inlet. If resistance is felt, unscrew the nut and retry. Excessive force can irreparably damage the inlet.

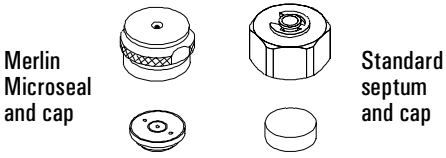
5. Tighten the retaining nut ½-turn past finger tight.
6. Check all connections for leaks. If necessary, the retaining nut can be tightened an additional ¼-turn to eliminate leaks.

Procedure: Changing the septum

Either a regular septum or a Merlin Microseal can be used with the septum head.

If the inlet temperature is set below 40°C, the Merlin Microseal may not seal effectively. For inlet temperatures below 40°C, use a regular septum for the inlet seal.

1. To replace the septum, cool the inlet to ambient temperature.
2. Using the inlet tool or manually, unscrew the septum cap or Merlin cap counterclockwise. If the septum head begins to turn, support it manually while removing the cap.
3. Remove the septum or Merlin Microseal, taking care not to scratch the interior of the septum head.
4. Install a new septum or Merlin Microseal and the correct cap. When installing a Merlin Microseal, note that the side where the metal parts are visible goes down.



5. Check for leaks out of the cap and tighten the cap if necessary.

Glass inlet liners

The liner is the chamber for sample deposition. Three kinds are available:

Table 50. Inlet liners

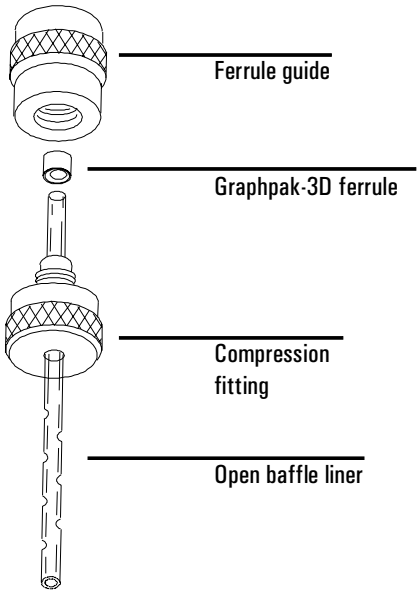
Type	Injection capacity	Inertness	Quantity	Part no.
Open baffled liner	Lowest capacity	Most inert	10	5182-9751
Liner packed with silanized glass wool	Higher capacity	Less inert	10	5182-9752
Unpacked liner, to be packed by the user	Depends on the packing		10	5182-9753

Type	Injection capacity	Glass type	Glass wool packing*	Typical application	Part no.
Single baffle liner	180 µL	Borosilicate deactivated	Yes	Large volume injection, not for extremely active compounds	5183-2038
Single baffle liner	200 µL	Borosilicate deactivated	No	General purpose	5183-2036
Multi baffle liner	150 µL	Borosilicate deactivated	No	Active compounds, drugs, pesticides	5183-2037
Fritted glass liner	150 µL	Borosilicate deactivated	No	Large volume injection, all but the most active compounds	5183-2041

*Silanized glass wool 10 gm (pesticide grade) part no. 5181-3317

Procedure: Replacing liners

1. Remove the head from the inlet. See "Procedure: Removing the septumless head" or "Procedure: Removing the septum head".
2. Grasp the liner by the Graphpak ferrule. Remove the liner and ferrule.
3. Unscrew the assembly tool (part number G2617-80540) into two pieces, the ferrule guide and the compression fitting.



4. Slide the compression fitting onto the longer straight end of the new liner with the threads pointing toward the end of the liner.
5. Place a Graphpak-3D ferrule on the same end of the liner with the recessed graphite end towards the compression fitting. Slide the ferrule on so that about 2 mm of liner is exposed beyond the ferrule.
6. Slide the compression fitting up to meet the ferrule. Screw the ferrule guide gently onto the compression fitting until it is finger tight.
7. Unscrew and remove the ferrule guide. Slide the compression fitting off the other end of the liner. The ferrule should now be set with 1 mm of liner exposed. Check that the graphite within the ferrule is flush with the top of the metal collar.

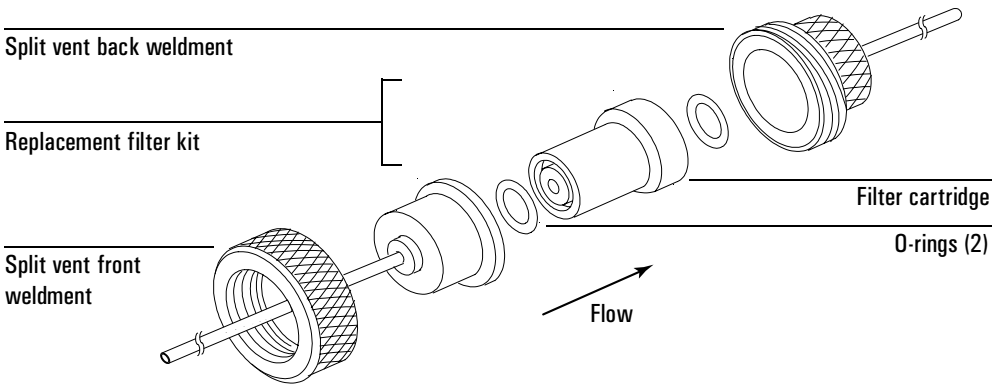
8. Insert the glass liner into the inlet from above until the unpacked side of the ferrule rests on the top of the inlet.
9. Replace the sampling head and reconnect the lines, if necessary.
10. Check all connections for leaks. If necessary, tighten them again by hand.

Replacing the split vent trap filter cartridge

WARNING Turn off the oven and turn off the heater for the inlet that uses the split vent trap and let them cool down. Turn off the carrier gas supply pressure.

The split vent trap may contain residual amounts of any samples or other chemicals you have run through the GC. Follow appropriate safety procedures for handling these types of substances while replacing the trap filter cartridge.

1. Turn off the inlet and the oven and allow to cool.
2. Set all GC flows to zero.
3. Remove the pneumatics cover.
4. Lift the filter trap assembly form the mounting bracket and unscrew the filter trap assembly.



5. Remove the old filter cartridge and O-rings and replace them.
6. Reassemble the trap.
7. Check for leaks.

Procedure: Leak testing the gas plumbing

Leaks in the gas plumbing can affect chromatographic results dramatically. The following procedure checks the flow system up to but not including the inlet flow manifold. If this portion of the system proves to be leak-free, refer to the next procedure to check the inlet and inlet manifold.

Liquid leak detectors are not recommended, especially in areas where cleanliness is very important.

If you do use leak detection fluid, immediately rinse the fluid off to remove the soapy film.

WARNING To avoid a potential shock hazard when using liquid detection fluid, turn the GC off and disconnect the main power cord. Be careful not to spill leak solution on electrical leads, especially the detector heater leads.

Materials needed:

- Electronic leak detector capable of detecting your gas type or liquid leak detection fluid. If you use leak detection fluid, remove excess fluid when you have completed the test.
- Two 7/16-inch wrenches

1. Using the leak detector, check each connection you have made, for leaks.
2. Correct leaks by tightening the connections. Retest the connections; continue tightening until all connections are leak-free.

Procedure: Leak testing the PTV inlet

There are numerous places in the inlet that can leak. This procedure lets you determine, in general, if there is an unacceptable leak in the inlet. If the inlet is leaking, you should use an electronic leak detector to pinpoint the component that is leaking.

WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

Materials needed:

- No-hole ferrule
- 7/16-inch wrench
- Gloves (if the inlet is hot)
- Septum nut wrench (part no. 19251-00100)
- 9/16-inch wrench
- 1/8-inch SWAGELOK cap
- Bubble flow meter

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Turn the oven off.
   - Cool the oven and inlet to room temperature.
   - Turn the inlet pressure off.
   - Remove the column, if one is installed, and plug the column fitting with the column nut and a no-hole ferrule.
   - Remove the old septum and replace it with a new one. For instructions, see "Procedure: Changing the septum".
2. Remove the column from the inlet fitting on the inside of the oven.
3. If a septum head is installed, and the quality of the septum (or Microseal) and Graphpak-3D ferrule on the glass liner are unknown, replace them now.

4. Cap the inlet's column fitting and the septum purge vent (septum head only). Use solid (no hole) Vespel type ferrules 1/8-inch (part no. 0100-1372) and 1⁄ 16-inch (part no. 5181-7458) with a 1/8-inch Swagelok nut (part no. 5180-4103) and a capillary column nut.

   As alternate capping devices, a 1/8-inch Swagelok cap can be used for the septum purge vent. A capillary column nut with a solid piece of wire the size of a paper clip and a 0.5 mm ID graphite ferrule may be used for the inlet column fitting.

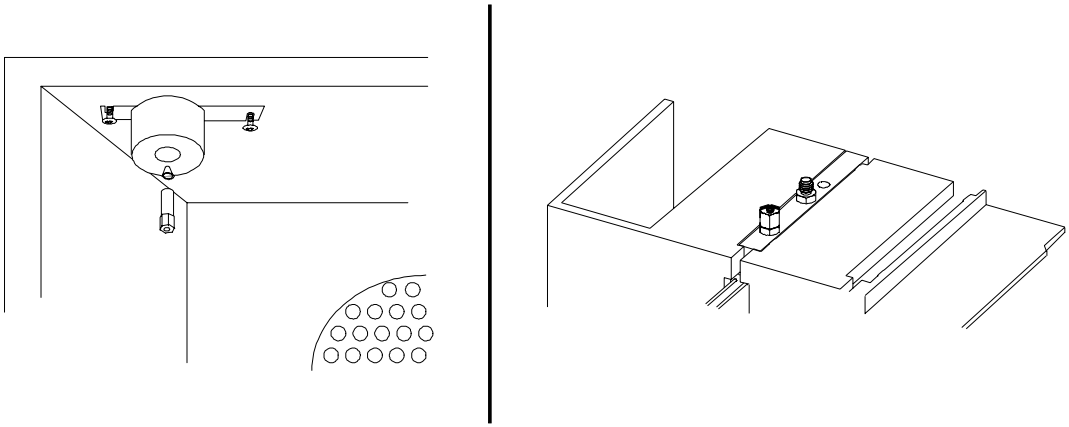


Figure 65 Capping the bottom of the inlet and septum purge vent

5. Make sure that the carrier gas source pressure is at least 35 psi. Carrier source pressure should always be at least 10 psi greater than the desired inlet pressure.

6. Configure the inlet for the test. Press `[Front Inlet]` (or `[Back Inlet]`) and:

   - Set the inlet to "Split Mode."
   - Configure the column as 0 length. Press `[Config] [Column 1]` or `[Config] [Column 2]` and enter "0" in the first column of the "Dim" field.
   - Set the inlet's Total Flow to 60 mL/min.

- Set the pressure to 25 psi.
- Set the inlet temperature to its normal operating temperature.

7. Wait approximately 15 seconds for equilibration.

   If pressure cannot be achieved, either a very large leak is present in the system, or the supply pressure is not high enough.

8. Turn the inlet pressure “Off.”

   Press `[Front Inlet]` (or `[Back Inlet]`), scroll to the “Pressure” field, and press `[Off]`. Both the flow controller and the back pressure valves will close.

9. Note the “Actual” reading on the display and monitor the pressure for 10 minutes.

   - If there is less than 0.5 psi pressure loss, consider the system leak tight.
   - If pressure loss is much greater than 0.5 psi, there is a leak that must be found and corrected. Note, however, that you may want to slightly decrease the leak test time based on the internal inlet volume which changes with the liner type used (smaller volumes = shorter acceptable leak test times). See “Correcting leaks” .

10. When the system is considered leak tight, the caps may be removed, the column reinstalled, its dimensions configured at keyboard, and the desired pressure and flow rate set.

Correcting leaks

Use an electronic leak detector to check all areas of the inlet and plumbing that are potential sources of a leak.

Tighten loose connections to correct leaks, if necessary. You may need to repeat the leak test.

If the pressure drop is now 0.5 psi or less, you can consider the inlet system leak–free. If the pressure drops faster than the acceptable rate, continue to search for leaks and repeat the pressure test.

Potential leak points

Check the following areas when checking an inlet system for leaks.

In the oven

- Make sure the bottom of the inlet is correctly capped.

On the inlet

- Septum (septum head only)
- Lower inlet seal at bottom of inlet
- Ferrule on inlet liner
- Connections for carrier gas, septum purge (septum head only)

At EPC module

- O-rings behind the block where the inlet's pneumatic lines enter the module
- Septum purge cap (septum head only)
- Chemical trap O-rings
- O-rings in gang fitting

Consumables and replaceable parts

Description	Quantity	Part no.
Septumless head assembly	1	G2617-60507
Service kit	1	5182-9747
Valve body	1	5182-9757
Pressure spring	1	5182-9758
Kalrez seal	1	5182-9759
Teflon guide	1	5182-9748
Sealing element	1	5182-9760
Graphpak-3D ferrule for liners	5	5182-9749
Assembly tool for Graphpak-3D ferrules	1	G2617-80540
Single baffle liner	1	5183-2038
Single baffle liner	1	5183-2036
Multi baffle liner	1	5183-2037
Fritted glass liner		5183-2041
Graphpak-2M inlet adapter, 0.2 mm column id	1	5182-9754
Graphpak-2M inlet adapter, 0.32/0.25 mm column id	1	5182-9761
Graphpak-2M inlet adapter, 0.53 mm column id	1	5182-9762
Silver seal for Graphpak-2M inlet adapter	5	5182-9763
Nut for Graphpak inlet adapters	5	5062-3525
Ferrules for Graphpak-2M inlet adapter, 0.2 mm column id	10	5182-9756
Ferrules for Graphpak-2M inlet adapter, 0.25 mm column id	10	5182-9768
Ferrules for Graphpak-2M inlet adapter, 0.32 mm column id	10	5182-9769
Ferrules for Graphpak-2M inlet adapter, 0.53 mm column id	10	5182-9770

more >

Description	Quantity	Part no.
Syringes		
5 µL, 23 gauge fixed needle	1	9301-0892
10 µL, 23 gauge fixed needle	1	9301-0713
10 µL, Teflon-tipped plunger, 23 gauge fixed needle	1	5181-8809
10 µL, Teflon-tipped plunger, 23 gauge removable needle	1	5181-8813
25 µL, Teflon-tipped plunger, 23 gauge fixed needle	1	5183-0316
25 µL, Teflon-tipped plunger, 23 gauge removable needle	1	5183-0317
50 µL, Teflon-tipped plunger, 23 gauge fixed needle	1	5183-0318
50 µL, Teflon-tipped plunger, 23 gauge removable needle	1	5183-0319
Septa and seals		
Merlin Microseal starter kit (cap + 1 microseal)	1	5182-3442
Merlin Microseal replacement	1	5182-3444
11 mm septa, red	25	5181-1263

18 The Volatiles Interface

Using a Volatiles Interface

Columns and Traps

Split mode

Understanding the pneumatics
Using the control table
Operating parameters
Split ratio
Procedure: Operating in the split mode with the column defined
Procedure: Operating in the split mode with the column not defined

Splitless mode

Understanding the pneumatics
Using the control table
Operating parameters
Procedure: Operating in the splitless mode

Direct mode

Understanding the pneumatics
Preparing your interface for direct sample introduction
Procedure: Disconnecting the split vent line
Procedure: Configuring for a direct injection
Using the control table
Operating parameters
Procedure: Operating in direct mode

Maintaining a Volatiles Interface

Procedure: Installing columns
Procedure: Replacing or cleaning the interface
Replacing the split vent trap filter cartridge
Procedure: Leak testing the gas plumbing
Procedure: Leak testing the system
Procedure: Preparing the interface for a leak test
Procedure: Correcting leaks

Connecting to an External Gas Sampler

Procedure: Connecting the 7694 headspace sampler
Procedure: Connecting the 7695 purge and trap concentrator

The Volatiles Interface

Using a Volatiles Interface

The volatiles interface provides a simple, reliable way to introduce a gas sample into your gas chromatograph (GC) from an external device such as the headspace, purge and trap, or air toxics samplers. The interface has a small volume and is highly inert, thus ensuring high sensitivity and resolution for applications requiring trace level detection.

Total flow to the interface is measured by a flow sensor and is divided into two streams. One stream connects to the septum purge regulator; the other connects to a frit block. At the frit block, the flow is further divided. The first stream goes to the gas-phase sampler and from there is introduced into the interface. The second stream, called the pressure sensing line, passes through the frit block and is measured by a pressure sensor. This stream also provides a trickle flow to the interface.

There are three modes of operation—split, splitless, and direct. The pneumatics vary for each operating mode and are discussed in detail in "Split mode" ,"Splitless mode" , and "Direct mode" . Table 51 summarizes some issues to consider when choosing an operating mode. Specifications for the interface are also listed.

Table 51 Overview of volatiles interface

Mode	Sample type (concentration)	Sample to column	Comments
Split	High	Very little, most is vented	
Splitless	Low	All	Can switch to split mode electronically.
Direct	Low	All	Must physically disconnect split vent, plug the interface, and reconfigure the GC. Maximizes sample recovery and eliminates possibility of contamination to pneumatic system.
Specifications			
Silcosteel®-treated flow path			
Volume:			32 µL
Internal dimensions:			2 mm by 10 mm
Maximum total flow to interface:			100 mL/min
Split range:			Dependent on column flow Typically no split to 100:1
Temperature range:			10° C above ambient (with oven at ambient) to 400° C
Recommended temperature:			≥ transfer line temperature of the external sampling device

Split mode

When you introduce a sample in the split mode, a small amount of the sample enters the column while the major portion exits from the split vent. The ratio of split flow to column flow is controlled by the user. The split mode is primarily used for high concentration samples when you can afford to lose most of the sample out the split vent and for samples that cannot be diluted.

Understanding the pneumatics

During Pre Run, during sampling, and after sampling, total flow to the interface is measured by a flow sensor and controlled by a proportional valve. Flow at the head of the column is back-pressure regulated. Pressure is sensed upstream from the proportional valve.

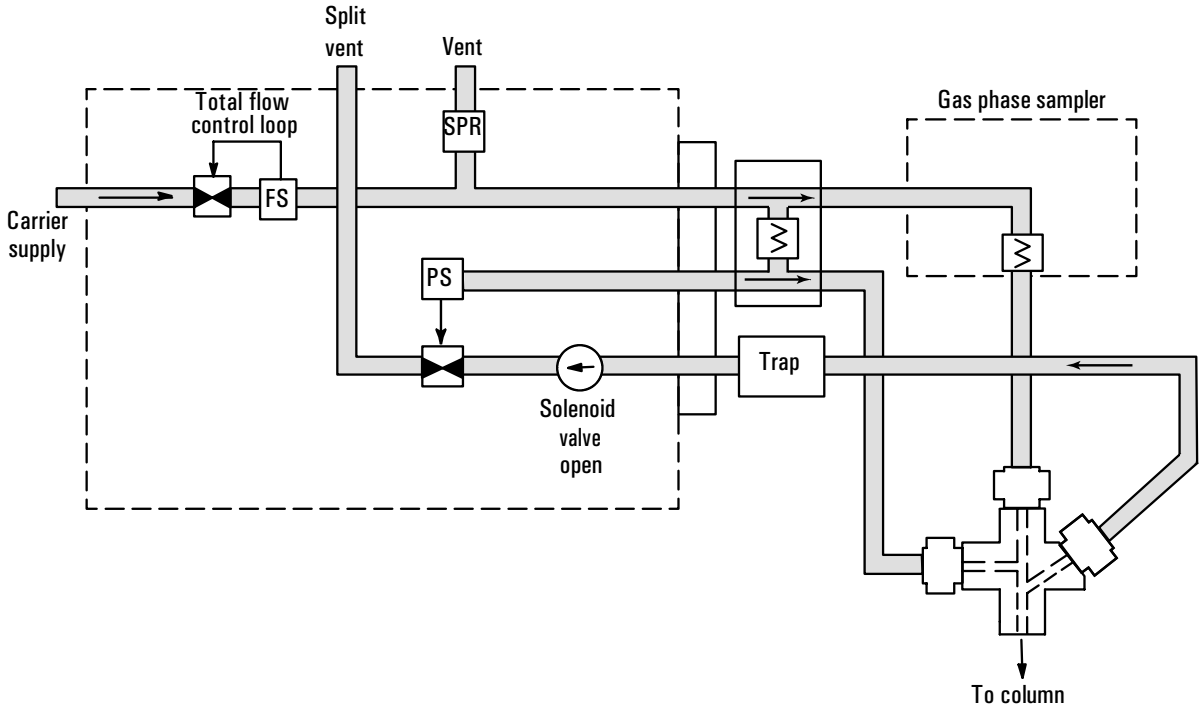


Figure 66 Pneumatics: Split mode
Splitless mode: Idle or after sampling end

Using the control table

`Mode:` The current operating mode—split

`Temp` Actual and setpoint interface temperatures

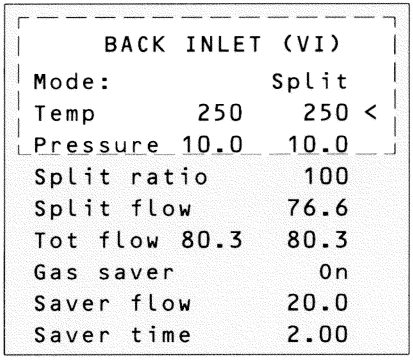
`Pressure` Actual and setpoint interface pressure

`Split ratio` The ratio of split flow to column flow. Column flow is set at the Column 1 or Column 2 control table. This parameter is not available if your column is not defined.

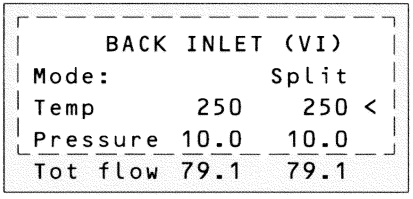
`Split flow` Flow, in mL/min, from the split vent. This parameter is not available if your column is not defined.

`Total flow` The total flow into the interface, both setpoint and actual.

Column defined



Column not defined



Some setpoints are interdependent. If you change one setpoint, other setpoints may change to compensate.

Table 52 Split mode pneumatic setpoints

Column defined	
When you change:	**These setpoints change:**
Pressure	Column flow*
	Split flow
	Total flow
Column flow*	Pressure
	Split flow
	Total flow
Split flow	Split ratio
	Total flow
Split ratio	Split flow
	Total flow
Total flow	Split flow
	Split ratio

*This setpoint appears in the column control table.

Column not defined

Setpoints for Column flow, Split flow, and Split ratio are not available.

You can change the setpoints for Total flow and Pressure without affecting other setpoints.

Operating parameters

Use the information in Table 53 to help you set up the operating conditions for your interface.

Table 53 Split mode operating parameters

Parameter	Allowed setpoint range	Suggested starting value
Oven initial time	0 to 999.9 minutes	After sample on column
Interface temperature	Ambient + 10° C to 400° C	≥ Transfer line temperature
Gas saver time	0 to 999.9 minutes	After sample on column
Gas saver flow	15 to 100 mL/min	15 mL/min greater than maximum column flow

Split ratio

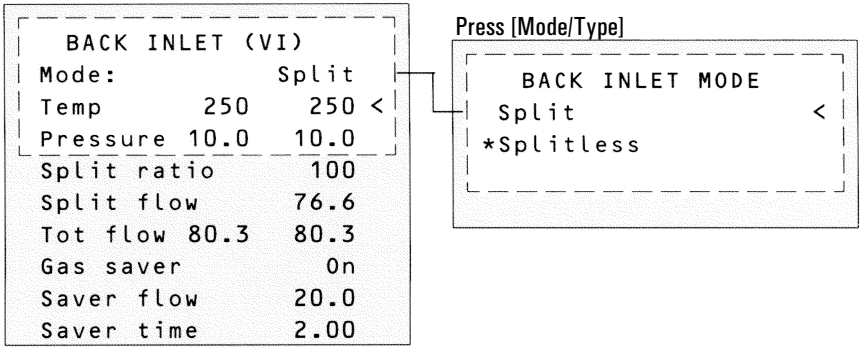
Because of the interface's small internal volume, the maximum total flow to the interface is 100 mL/min. This maximum flow puts some restriction on the split ratio you can set.

Table 54 Split ratio

Column diameter (μm)	Column flow (mL/min)	Maximum split ratio	Total flow (mL/min)
200	1	100:1	100
530	5	20:1	100

Procedure: Operating in the split mode with the column defined

1. Verify that the split vent line is connected to your interface. Verify that the [Config][Inlet] control table displays “split plumbed”.
2. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See “Flow and Pressure Control”.
3. Press [Front Inlet] or [Back Inlet].



   a. Scroll to `Mode`: and press [Mode/Type]. Select `Split`.

Split ratio = Split flow / Column flow

   b. Set the interface temperature.
   c. If you want a specific split ratio, scroll to `Split ratio` and enter that number. The split flow will be calculated and set for you.
   d. If you want a specific split flow, scroll to `Split flow` and enter that number. The split ratio will be calculated and set for you.
   e. If desired, turn on `Gas saver`. Set the `Saver time` after the sample has been introduced.
   f. If gas saver is on, be certain `Auto prep run` is On (see page 285) or use the [Prep Run] key before introducing the sample.

Procedure: Operating in the split mode with the column not defined

1. Verify that the split vent is connected to your interface. Verify that the [Config][Inlet] control table displays “split plumbed”.
2. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See “Flow and Pressure Control”.

3. Press [Front Inlet] or [Back Inlet].

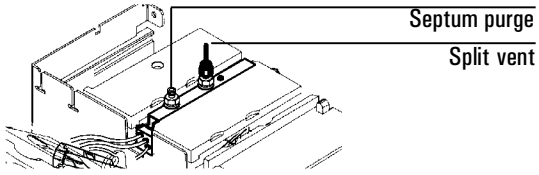
```
      BACK INLET (VI)
Mode:              Split
Temp        250     250 <
Pressure  10.0     10.0
Tot flow  79.1     79.1
```

   a. Set the temperature.

   b. Set total flow into the interface. Measure flow out of the split vent using a flow meter.

   c. Subtract the split vent flow from the `Total flow`. Subtract the septum purge flow (see "Septum purge" for nominal septum purge flows).

   d. Calculate the split ratio. Adjust as needed.



Front of GC

Splitless mode

When you introduce a sample, the solenoid valve remains closed while the sample enters the interface and is transferred to the column. At a specified time after the sample is introduced, the solenoid valve opens.

Understanding the pneumatics

Before Pre Run, when the GC is preparing for sample introduction, total flow to the interface is measured by a flow sensor and controlled by a proportional valve. Column flow is controlled via back-pressure regulation. See Figure 67.

During sampling, pressure upsets caused by switching valves in the external sampling device can cause fluctuations in column flow rates. To compensate for this, the interface is flow controlled during sampling time. The sampling flow rate is calculated from the pressure setpoint that is active when sample introduction begins. This flow control starts when the GC goes into the Pre Run state (when your system is automated and the Pre Run light is on or during manual operation when you press [Prep Run]) and ends after the interface's `Sampling end` setpoint expires.

During this user-specified sampling period, the solenoid valve is closed. Flow to the interface is measured by a flow sensor and controlled by a proportional valve. See Figure 67.

After sampling end, the solenoid valve opens. Flow to the interface is again measured by a flow sensor and controlled by a proportional valve while column flow is controlled via back-pressure regulation. The purge flow is controlled by the user. If desired, gas saver can be turned on at the end of the run. See Figure 67.

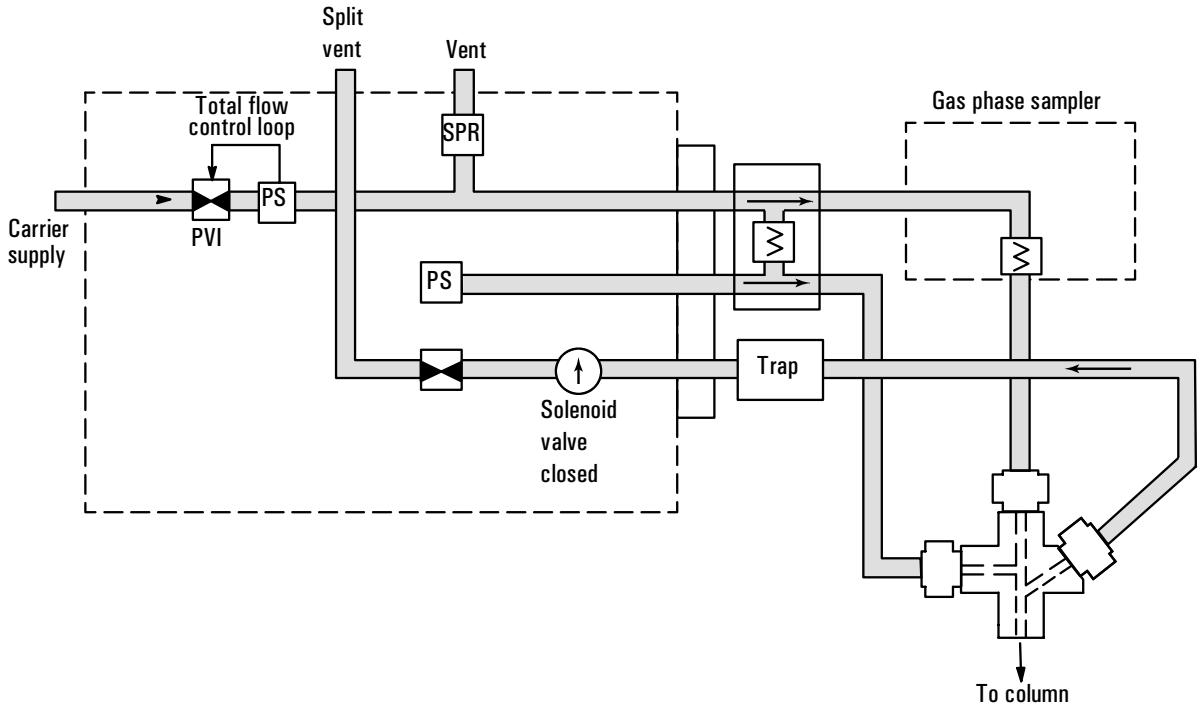


Figure 67 Splitless mode pneumatics: beginning of pre run to sampling end *(sample introduction in progress)*

Using the control table

`Mode`: The current operating mode—splitless

`Temp` Actual and setpoint interface temperatures

`Sampl'g end` The sample introduction interval, in minutes. The flow rate is calculated from the pressure setpoint that is active at the start of sample introduction.

Set the sampling end setpoint 0.2 minutes longer than the time the sampler needs to introduce the sample. For example, the 7694 headspace sampler has an Inject time parameter which controls how long the valve remains in the inject position. If Inject time is 1 minute, the sampling end setpoint should be set to 1.2 minutes.

If you're using an 7695 Purge and Trap Concentrator, set the Sampling end setpoint 0.2 minutes longer than the Desorb time parameter.

If your column is defined and you specify a flow or pressure program for your column, the ramp does not begin until after the sampling end setpoint expires.

`Pressure` Actual and setpoint interface pressure in psi, bar, or kPa.

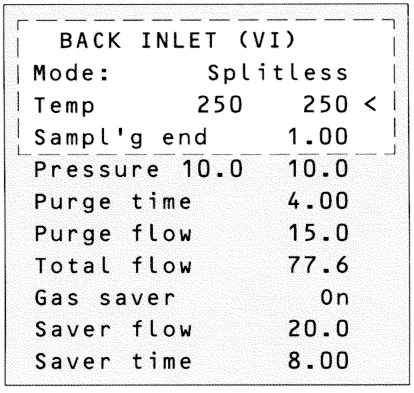
`Purge time` The time, after the beginning of the run, when purging resumes.

`Purge time` must be greater than `Sampling end`.

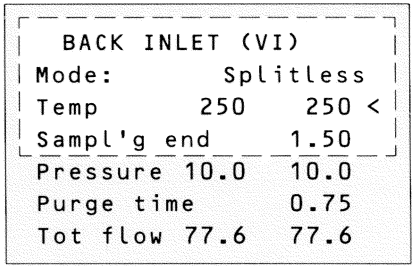
`Purge flow` The flow, in mL/min, from the split vent at `Purge time`. You will not be able to access or specify this value if operating with your *column not defined*.

`Total flow` When your column is defined, `Total flow` displays the actual flow to the interface. You cannot enter a setpoint. If your column is not defined, `Total flow` will have both setpoint and actual values during purge time. All other times, the actual flow to the interface is displayed.

Column defined



Column not defined



Some setpoints in the flow system are interdependent. If you change one setpoint, other setpoints may change to compensate.

Table 55 Splitless Mode Pneumatic Setpoints

Column defined	
When you change:	**These setpoint change:**
Purging	
Purge flow	Total flow**
Pressure	Total flow** Column flow*
Column flow*	Pressure Total flow**
Before and after sampling, not purging	
Pressure	Column flow* Total flow**
Column flow*	Pressure Total flow**
During sampling: You cannot change pressure and flow setpoints during sampling time.	

*This setpoint appears in the column control table.
**This value is actual only.

Column not defined
Purging: You can change the Pressure and Total flow setpoints; other setpoints are not affected.
Before and after sampling, not purging: You can change the Pressure setpoint; other setpoints are not affected.
During sampling: You cannot change pressure and flow setpoints during sampling time.

Operating parameters

A successful splitless injection consists of these steps:

1. Introduce a gas sample into the heated interface.
2. Use a low oven temperature while the sample collects at the head of the column.
3. Set your sampling end time to allow the entire sample to be swept out the sampler.
4. Set the purge time so that all the sample has collected on the column.
5. Begin your oven temperature program.

Table 56 Splitless Mode Operating Parameters

Parameter	Allowed setpoint range	Suggested starting value
Oven initial time	0 to 999.9 minutes	≥ Interface purge time
Interface temperature	Ambient + 10° C to 400° C	≥ Transfer line temperature
Interface sampling end	0 to 999.9 minutes	0.2 Minutes longer than introduction time
Interface purge time	0 to 999.9 minutes	
Gas saver time	0 to 999.9 minutes	Must be after purge time
Gas saver flow	15 to 100 mL/min	15 mL/min greater than maximum column flow

Procedure: Operating in the splitless mode

These instructions apply to both column *defined* and *not defined*.

1. Verify that the split vent line is connected to your interface. Verify that the [Config][Inlet] control table displays “split plumbed”.
2. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
3. Press [Front Inlet] or [Back Inlet].
   a. Scroll to `Mode`: and press [Mode/Type]. Select `Splitless`.
   b. Set the interface temperature and a sampling end time.

Column defined

```
  BACK INLET (VI)
Mode:       Splitless
Temp       250    250 <
Sampl'g end        1.5
Pressure 10.0     10.0
Purge time        1.75
Purge flow        15.0
Total flow        77.6
Gas saver           On
Saver flow        20.0
Saver time        2.00
```

Column not defined

```
  BACK INLET (VI)
Mode:       Splitless
Temp       250    250 <
Sampl'g end       1.50
Pressure 10.0     10.0
Purge time        0.75
Tot flow 77.6     77.6
```

If using gas saver, set time after purge flow time.

   c. If your column is defined, enter a purge time and purge flow. Turn Gas saver on if desired. Set the `Gas saver` time *after* the purge time and enter a Gas saver flow.
   d. If your column is not defined, enter a purge time (purge flow is not available). Set total flow greater than column flow plus septum purge flow (about 6 mL/min) to guarantee adequate column flow.
4. Make certain `Auto Prep Run` is On (see page 285) or use the [Prep Run] key before introducing a sample.

Direct mode

Direct sample introduction permits a quantitative transfer of analyte without risking contamination to the pneumatic system. It provides the sensitivity required for air toxics analyses. The interface's minimal dead volume also eliminates the potential interaction of solutes with poorly swept, active surfaces.

To operate in the direct mode, you must physically disconnect the split vent and reconfigure the GC. Instructions for performing these procedures are discussed in "Connecting to an External Gas Sampler".

Understanding the pneumatics

Before Pre Run, the interface is forward pressure controlled; pressure is sensed downstream from the flow proportional valve. See Figure 68a.

During sampling, pressure upsets caused by switching valves in the external sampler can cause fluctuations in column flow rates. To compensate for this, the interface is flow controlled during sampling time. The sampling flow rate is calculated from the pressure setpoint that is active when sample introduction begins. This flow control starts when the GC goes into the Pre Run state (when your system is automated and the Pre Run light is on or during manual operation when you press [Prep Run]) and ends after the interface's `Sampling end` setpoint expires.

Flow to the interface is measured by a flow sensor and controlled by a proportional valve. See Figure 68b.

After sampling end, the interface is forward pressure controlled; pressure is sensed downstream from the proportional valve. See Figure 68a.

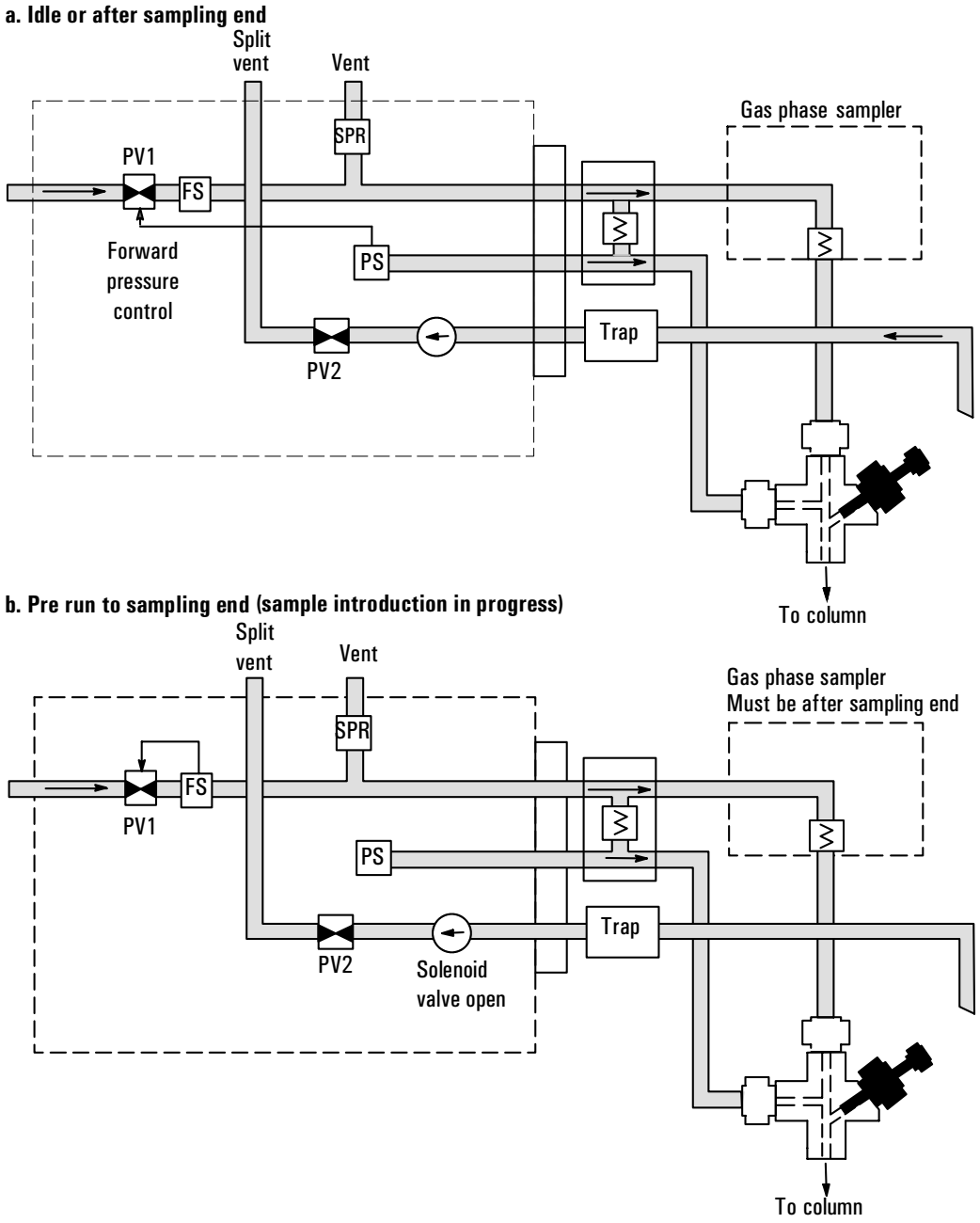


Figure 68 Pneumatics for direct mode

Preparing your interface for direct sample introduction

Before you can operate your interface in direct mode, you must:

- Disconnect the split vent line
- Configure the GC for a direct injection

Procedure: Disconnecting the split vent line

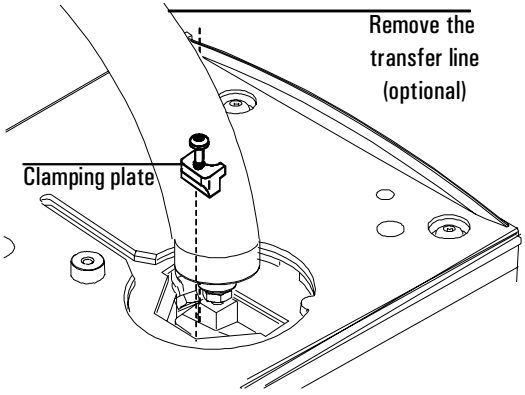
WARNING Be careful! The interface may be hot enough to cause burns.

Materials needed:

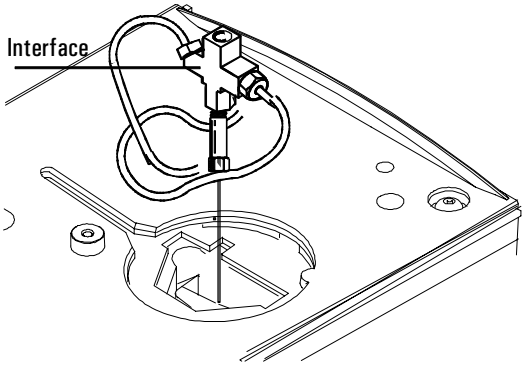
- Blanking nut
- 1/4-inch wrench
- 5/16-inch or adjustable wrench
- T-20 Torx screwdriver

1. Press [Front Inlet] or [Back Inlet] and turn off the interface temperature and pressure. Allow the interface to cool.
2. If desired, remove the transfer line by loosening the hex nut with a 1/4-inch wrench. Remove the clamping plate from the interface by

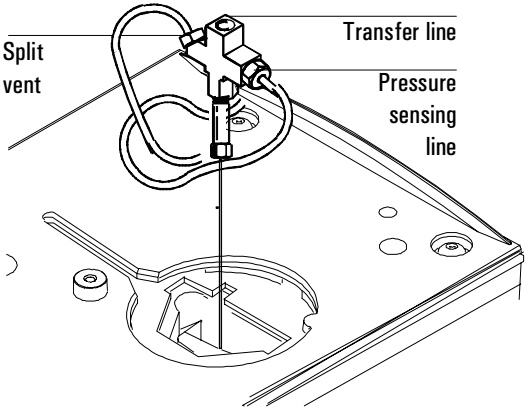
   loosening the captive screw with a T-20 Torx screwdriver. Put the plate in a safe place.



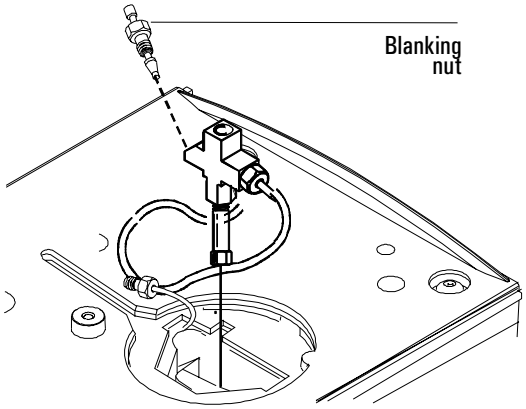
3. Carefully lift the interface out of the heater block.



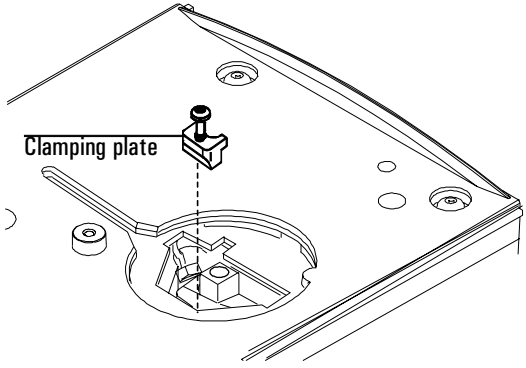
4. Loosen the hex nut connecting the split vent line to the interface until you can remove the line. Put the line aside. You do not need to plug it.



5. Install a blanking nut into the split line port and finger tighten the nut. Tighten the nut an additional 1/4-turn using two wrenches in opposition, the adjustable wrench on the interface and the 1/4-inch wrench on the nut.



6. Place the interface in the heater block. Replace the clamping plate you removed in Step no. 2 and tighten the screw until snug. Do not overtighten. If you removed the transfer line, replace it.

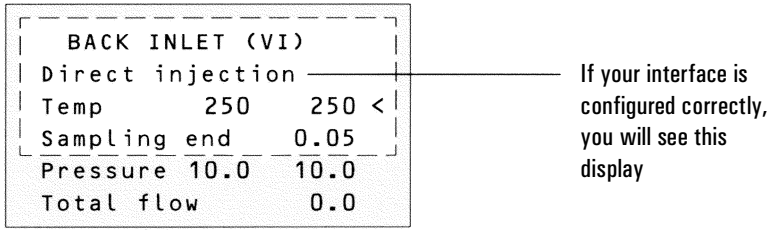


7. Restore the GC to normal operating conditions. Perform a leak test on the interface fittings.

Procedure: Configuring for a direct injection

The GC cannot sense the presence of the split vent. When you disconnect or reconnect the vent, you must configure the GC so that the pneumatics work properly.

1. Press [Config] [Back Inlet] or [Config] [Front Inlet].
2. Press [Mode/Type].
3. Choose `Split removed`.
4. Press [Back Inlet] or [Front Inlet]. If your GC is correctly configured, you will see the following display:



Using the control table

`Direct injection` If your GC is configured correctly, you will see above display.

`Temp` Actual and setpoint interface temperatures

`Sampl'g end` The sample introduction interval, in minutes. The flow rate is calculated from the pressure setpoint that is active at the start of sample introduction.

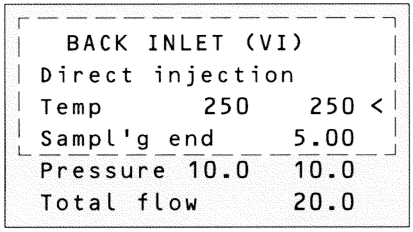
Set the sampling end setpoint 0.2 minutes longer than the time the sampler needs to introduce the sample. For example, the 7694 headspace sampler has an Inject time parameter which controls how long the valve remains in the inject position. If Inject time is 1 minute, the sampling end setpoint should be set to 1.2 minutes. If you're using an 7695 Purge and Trap Concentrator, set the Sampling end setpoint 0.2 minutes longer than the Desorb time parameter.

If your column is defined and you specify a flow or pressure program for your column, the ramp does not begin until after the sampling end setpoint expires.

`Pressure` Actual and setpoint interface pressure before a run and after sampling time.

`Total flow` The actual flow to the interface. This is a reported value, not a setpoint.

Column defined or column not defined



Some setpoints in the flow system are interdependent. If you change one setpoint, other setpoints may change to compensate.

Table 57 Direct Mode Pneumatic Setpoints

Column defined	
When you change:	**These setpoints change:**
Before and after sampling	
Pressure	Column flow* Total flow**
Column flow*	Pressure Total flow**
During sampling	
You cannot change pressure and flow setpoints during sampling time.	
Column not defined	
Before and after sampling	
The Column flow* setpoint is not available.	
You can change the pressure setpoint; other setpoints are not affected.	
During sampling	
You cannot change pressure and flow setpoints during sampling time.	

*This setpoint appears on the column control table.
**This value is actual only.

Operating parameters

Use the information in Table 58 to help you set up the operating conditions for your interface.

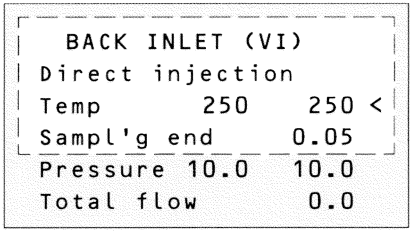
Table 58 Direct Mode Operating Parameters

Parameter	Allowed setpoint range	Suggested starting value
Oven initial time	0 to 999.9 minutes	≥ interface sampling end
Interface temperature	Ambient + 10° C to 400° C	≥ transfer line temperature
Interface sampling end	0 to 999.9 minutes	0.2 minutes longer than actual sampling time

Procedure: Operating in direct mode

These instructions apply to both column *defined* and *not defined*.

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control" .
2. Press [Front Inlet] or [Back Inlet].
   a. Verify that your GC is configured for a direct injection.
   b. Set the interface temperature.
   c. Set sampling end. Set 0.2 minutes longer than the sample introduction time.



3. Make certain `Auto Prep Run` is On (see page 285) or use the [Prep Run] key before introducing a sample.

Maintaining a Volatiles Interface

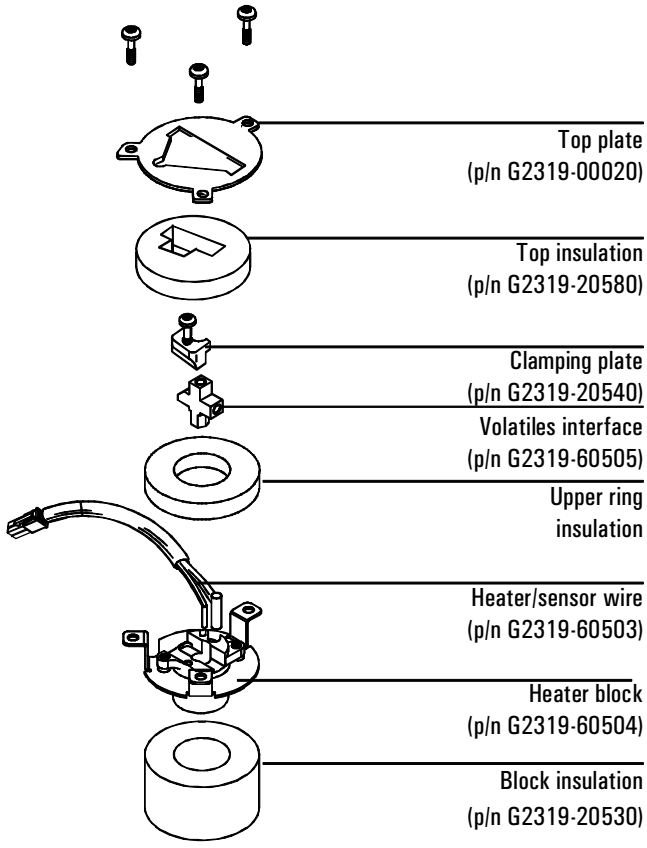


Figure 69 The volatiles interface parts breakdown

Procedure: Installing columns

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

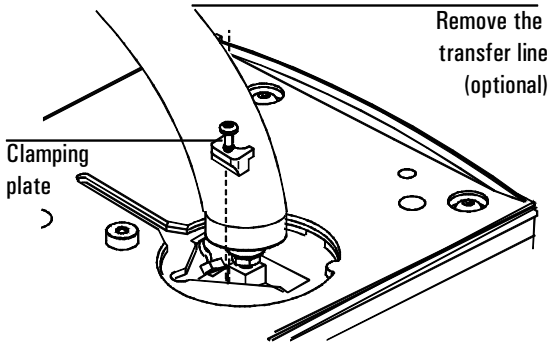
WARNING Be careful! The interface may be hot enough to cause burns.

Materials needed**:**

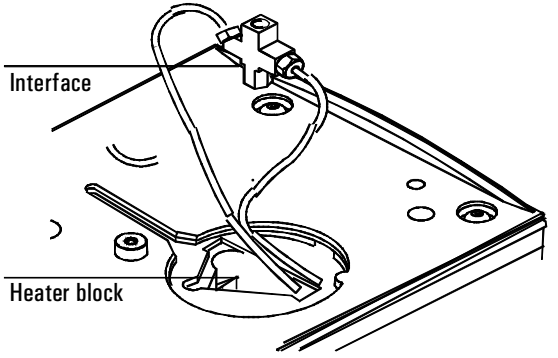
- Column nut and ferrule
- Column cutter
- Tissue
- Typewriter correction fluid
- 1/4-inch wrench
- 5/16-inch or adjustable wrench
- Metric ruler
- T-20 Torx screwdriver

1. Press [Oven] and set the oven to 35°C. Press [Front Inlet] or [Back Inlet] and turn off the interface temperature and pressure. Allow the interface to cool. When the oven temperature reaches setpoint, turn the oven off.
2. Disconnect the transfer line, if desired. Loosen the nut with a 1/4-inch wrench and remove the line. Remove the clamping plate from the interface

by loosening the captive screw with a T-20 Torx screwdriver. Put the plate in a safe place.

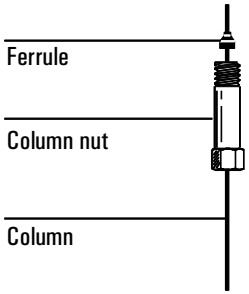


3. Lift the interface out of the heater block.

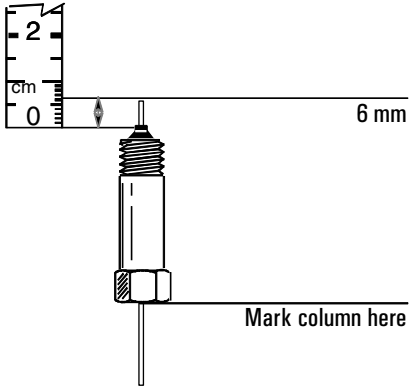


4. From inside the oven, push the column through the opening in the oven top. Grab the column from the oven top.

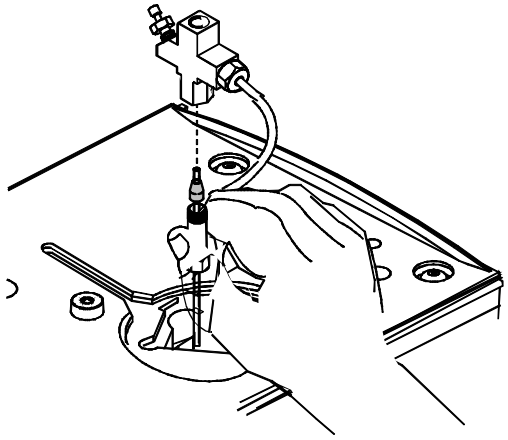
5. Place a capillary column nut and ferrule on the column and prepare the column end. If you need help with this step, "Procedure: Preparing capillary columns".



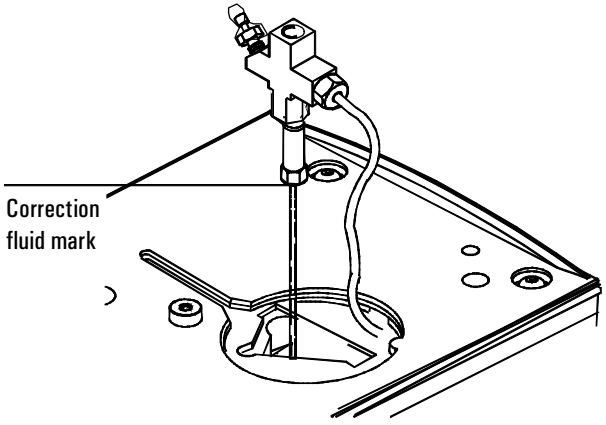
6. Position the column so it extends 6 mm above the end of the ferrule. Mark the column with typewriter correction fluid at a point even with the column nut.



7. Insert the prepared column in the interface and finger tighten the column nut.

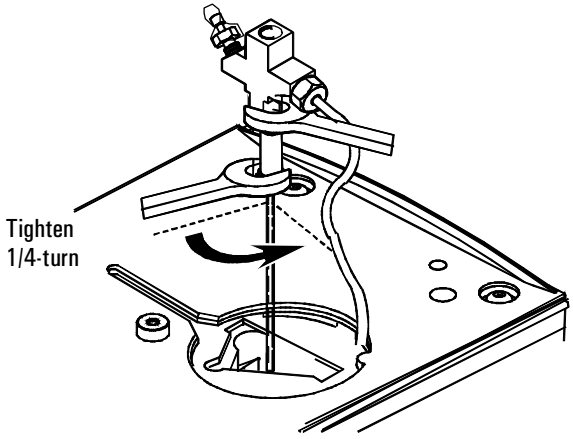


8. Adjust the column position so that the correction fluid mark on the column is even with the bottom of the column nut.

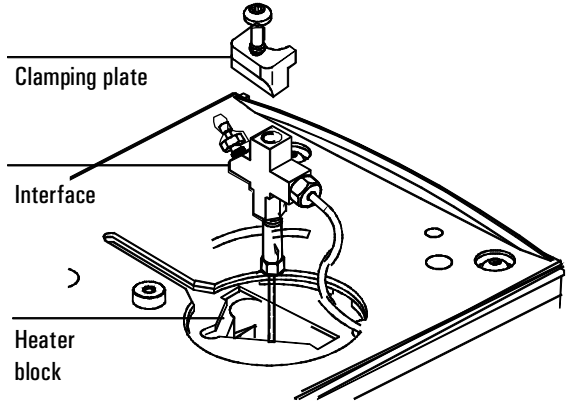


9. Tighten the column nut an additional 1/4- to 1/2-turn. Use the adjustable wrench to hold the interface while you tighten the column nut with the

1/4-inch wrench until the column cannot be pulled from the fitting with gentle pressure.



10. Replace the interface in the heater block. Replace the clamping plate and tighten the screw until snug. If you removed the transfer line, reinstall it.



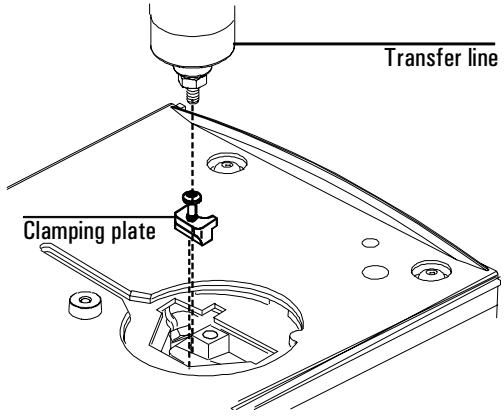
11. After the column is installed at both interface and detector, establish a flow of carrier gas through the interface. Heat the interface to operating temperature. Retighten the fittings, if necessary.

Procedure: Replacing or cleaning the interface

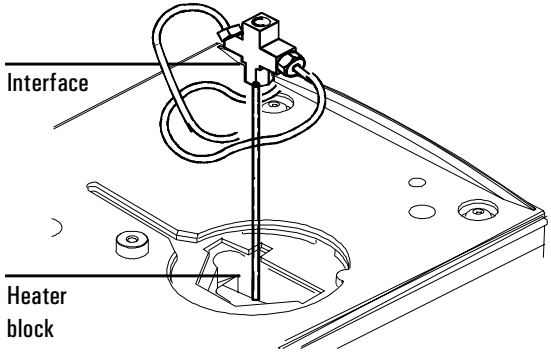
Materials needed:

- 1/4-inch or 7-mm wrench
- Sonicator or new interface
- T-20 Torx screwdriver

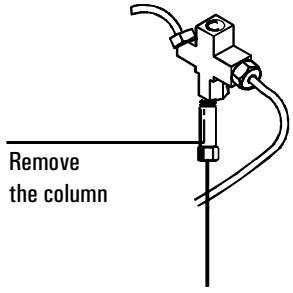
1. If you have entered parameters that you do not want to lose, store them as a method. Allow the oven and interface to cool. Turn off all flows at the initial gas supply or set the flows to 0 in the inlet control table.
2. Disconnect the transfer line. Loosen the nut with a 1/4-inch wrench and remove the line. Remove the clamping plate from the interface by loosening the captive screw with a T-20 Torx screwdriver. Put the plate in a safe place.



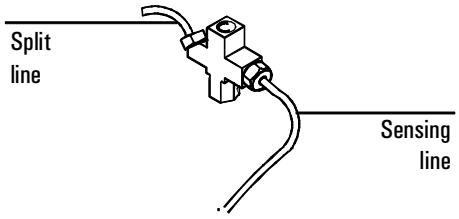
3. Lift the interface out of the heater block.



4. If a column is installed, remove it.



5. Remove the split and pressure sensing lines by loosening the hex nuts with the wrench.

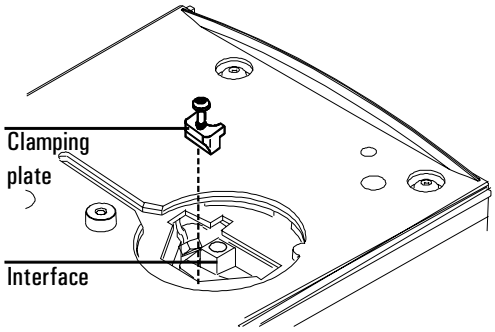


6. Clean or replace the interface. If you are cleaning the interface, sonicate it twice and then rinse.

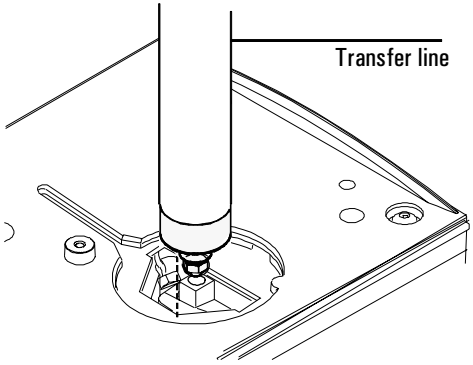
   Reinstall the split line and pressure sensing lines and finger tighten the hex nuts. Tighten the hex nuts an additional 1/4-turn with the wrench.

7. Reinstall the column in the interface. See "Procedure: Installing columns".

8. Place the interface in the heater block. Replace the clamping plate you removed earlier and tighten the screw until snug. Do not overtighten.



9. Reinstall the transfer line. Finger tighten the nut and then tighten an additional 1/4-turn with the wrench.



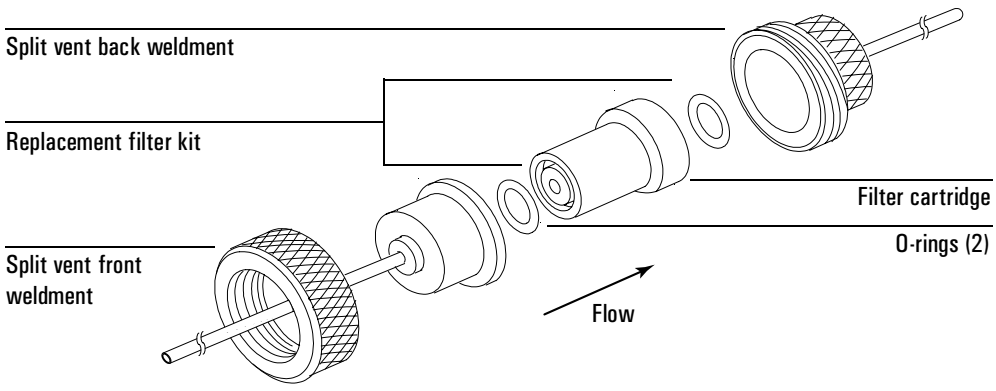
10. After the column is installed at both interface and detector, establish a flow of carrier gas through the interface and maintain it for 10 to 15 minutes. Check for leaks. Heat the interface to operating temperature and retighten the fittings, if necessary.

Replacing the split vent trap filter cartridge

WARNING Turn off the oven and turn off the heater for the inlet that uses the split vent trap and let them cool down. Turn off the carrier gas supply pressure.

The split vent trap may contain residual amounts of any samples or other chemicals you have run through the GC. Follow appropriate safety procedures for handling these types of substances while replacing the trap filter cartridge.

1. Turn off the inlet and the oven and allow to cool.
2. Set all GC flows to zero.
3. Remove the pneumatics cover.
4. Lift the filter trap assembly form the mounting bracket and unscrew the filter trap assembly.



5. Remove the old filter cartridge and O-rings and replace them.
6. Reassemble the trap.
7. Check for leaks.

Procedure: Leak testing the gas plumbing

Leaks in the gas plumbing can affect chromatographic results dramatically. The following procedure checks the flow system up to but not including the interface flow module. If this portion of the system proves to be leak-free, refer to the next procedure to check the interface and interface module.

Liquid leak detectors are not recommended, especially in areas where cleanliness is very important. If you do use leak detection fluid, immediately rinse the fluid off to remove the soapy film.

WARNING To avoid a potential shock hazard when using liquid detection fluid, be careful not to spill leak solution on electrical leads, especially the detector heater leads.

Materials needed:

- Electronic leak detector capable of detecting your gas type or liquid leak detection fluid. If you use leak detection fluid, wipe off excess fluid when you have completed the test.
- Two 7/16-inch wrenches

1. Using the leak detector, check each connection you have made for leaks.
2. Correct leaks by tightening the connections with the wrenches. Retest the connections; continue tightening until all connections are leak-free.

Procedure: Leak testing the system

There are several places in the interface-sampler system that can leak. This procedure helps you determine, in general, if there is an unacceptable leak in the system. If there is a leak, you should use an electronic leak detector to pinpoint the component that is leaking.

WARNING Be careful! The oven and interface may be hot enough to cause burns.

Materials needed:

- No-hole ferrule
- 7/16-inch wrench
- Two, 1/8-inch SWAGELOK caps
- Gloves (if the interface is hot)
- 1/4-inch or 7 mm wrench

1. Complete the following preliminary steps:
   a. If you have entered parameters that you do not want to lose, store them as a method.
   b. Cool the oven to room temperature and then turn it off.
   c. When the oven is cool, turn off the interface pressure from the keyboard.
   d. Remove the column, if one is installed, and plug the column fitting with the column nut and a no-hole ferrule.
2. Cap the septum purge and split vent fittings located on the flow module with 1/8-inch Swagelok caps.
3. Press [Front Inlet] or [Back Inlet] to open the control table. Enter a pressure setpoint between 20 and 25 psi, or enter your normal operating pressure if it is greater. Make sure that the pressure at the initial gas supply is at least 10 psi higher than the interface pressure. Wait a few minutes for the pressure to equilibrate.
4. Turn the pressure off. Because the septum purge, split vent, and column fittings are capped, gas should be trapped in the system and the pressure

should remain fairly constant. Turn the pressure off at the source if you want to isolate the pneumatic system completely.

5. Continue to monitor pressure for 10 to 15 minutes. The pressure should drop approximately 1 psi during the first 1 to 2 minutes. After an initial pressure drop of about 1 psi, the pressure should not drop more than 0.03 psi/min.

   If the pressure drop is 0.03 psi/min or less, you can consider the interface-gas sampler system leak-free.

   If the pressure drops faster than the acceptable rate, you must check the interface and sampler systems separately to determine the source of the leak. See "Procedure: Preparing the interface for a leak test" to create a closed flow system, then return to this section and complete Steps 3 to 5 again.

   If you find a leak in the interface, refer to "Procedure: Correcting leaks".

   If the interface is leak-free, pressure check the sampling device. See the operating manual for your sampler for instructions.

Procedure: Preparing the interface for a leak test

To leak check the interface independent of the gas sampling device, you must disconnect the sampler from the interface to isolate the interface flow system from the sampler.

WARNING Be careful! The oven and interface may be hot enough to cause burns.

Materials needed:

- 1/16-inch male GC nut
- Graphite/Vespel ferrule

1. Disconnect the transfer line from the interface (see page 478).
2. Disconnect the carrier line from the sampler (see page 479 if you have a Headspace sampler or page 483 if you have a Purge and Trap Concentrator).
3. Prepare the end of the carrier line using the 1/16-inch male GC nut and the graphite/vespel ferrule.
4. Connect the carrier line to the interface where you removed the transfer line and tighten the nut finger tight and then tighten 1/4 to 1/2 turn with the 1/4-inch wrench.
5. Return to “Leak testing the system” and repeat steps 3 to 5.

Procedure: Correcting leaks

Materials needed:

- Electronic leak detector
- Tool that will tighten leaking fittings — 1/4-inch, 5/16-inch, or 7-mm wrench

1. Use the electronic leak detector to check all areas of the interface that are potential sources of a leak. Potential leak areas are:
   - The capped purge vent
   - The capped split vent
   - The plugged column connection
   - The area where the gas lines are plumbed to the interface
2. Correct leaks using the correct size wrench to tighten connections. You may need to repeat the leak test again to check for leaks.

   If the pressure drop is now 0.03 psi/min or less, you can consider the interface system leak-free.

   If the pressure drops faster than this, continue to search for leaks and repeat the pressure test. If all fittings appear to be leak-free but the interface system is still losing too much pressure, you may need to replace the interface module. Contact your Agilent service representative.

Connecting to an External Gas Sampler

Figure 70 shows a gas sampling device connected to the volatiles interface.

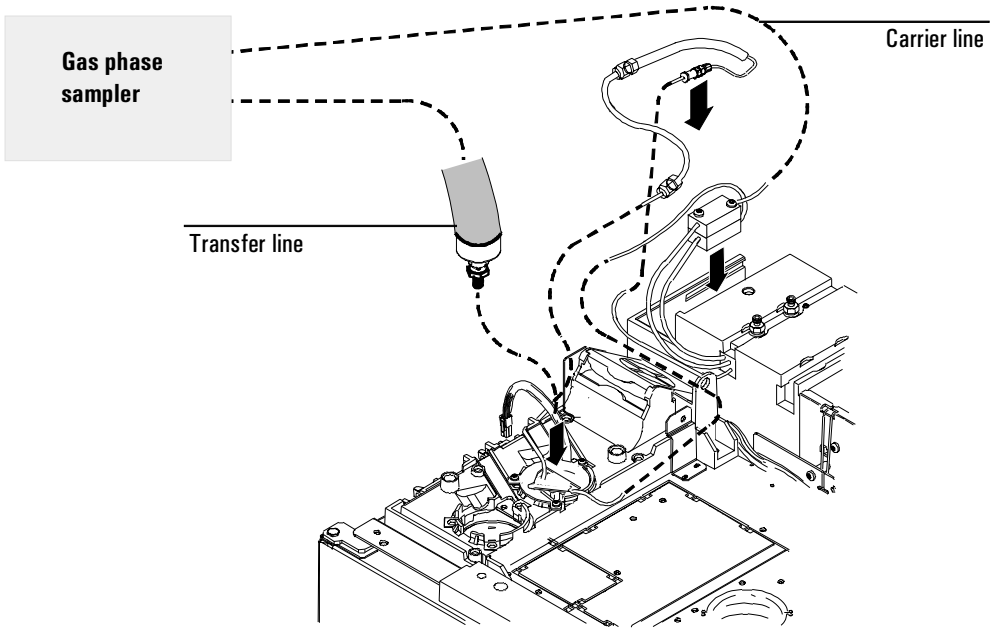


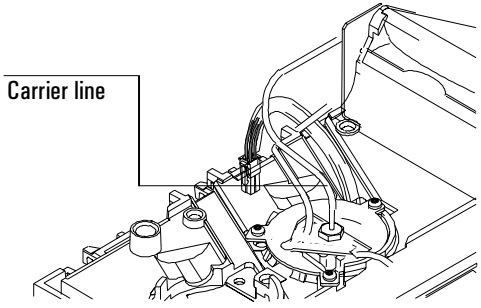
Figure 70 Flow diagram of an external sampling device

Procedure: Connecting the 7694 headspace sampler

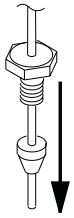
Materials needed:

- 1/8-inch Swagelok nut
- 1/16-inch to 1/8-inch reducer
- 1/8-inch ferrule set
- Wrenches
  — One 7/16-inch
  — Two 5/16-inch
  — One 1/4-inch
  — One 7-mm

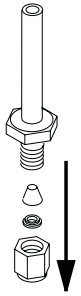
1. Remove carrier line tubing labeled “supply” attached to the volatiles interface using a 1/4-inch wrench.



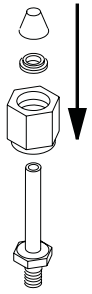
2. Remove the male fitting and Vespel/graphite ferrule from the carrier line. Keep the parts for later use.



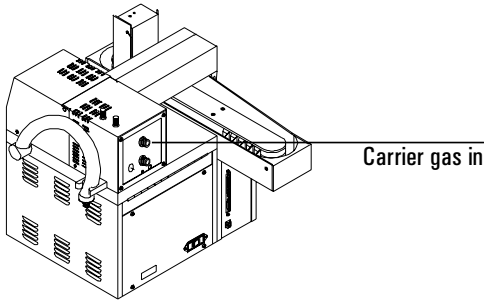
3. Remove the nut and the metal ferrules from a 1/16-inch to 1/8-inch reducer. Keep the parts for later use.



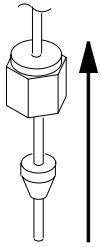
4. Slide a 1/8-inch Swagelok nut, a 1/8-inch back ferrule, and a 1/8-inch front ferrule onto the unthreaded end of the reducer.



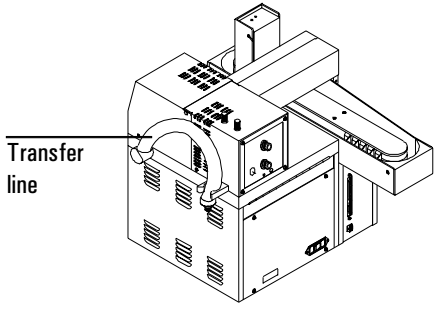
5. Connect the reducer to the gas supply port labeled “Carrier” on the back of the headspace sampler by tightening the 1/8-inch Swagelok nut using a 7/16-inch wrench. Tighten the nut 1/4-turn past finger tight.



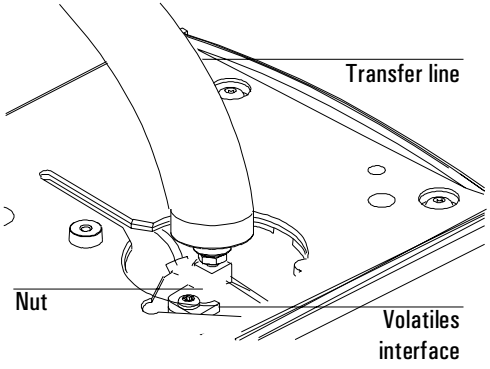
6. Slide the 1/16-inch female nut from step 3 and then the 1/16-inch Vespel/graphite ferrule from step 2 onto the end of the carrier line.



7. Connect the carrier line to the gas supply port. Use two wrenches to tighten the 1/16-inch Swagelok nut 1/4-turn past finger tight. Do not overtighten. If the fitting leaks, tighten an additional 1/8-turn.
8. Locate the headspace sampler's transfer line tubing.



9. Connect the transfer line (with the pre-attached nut and steel ferrule) to the interface. Tighten the nut 1/4-turn past finger tight. Do not overtighten. If the nut leaks, tighten an additional 1/8-turn.

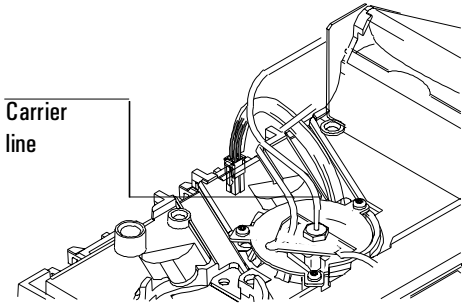


Procedure: Connecting the 7695 purge and trap concentrator

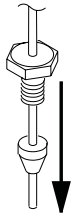
Materials needed:

- 1/16-inch Swagelok nut
- Vespel/graphite ferrule of the appropriate size for the transfer line
- Column cutter (fused silica)
- 5/16-inch and 1/4-inch wrenches
- Typewriter correction fluid
- Metric ruler

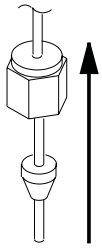
1. Remove the GC carrier line tubing labeled “supply” attached to the volatiles interface using a 1/4-inch wrench.



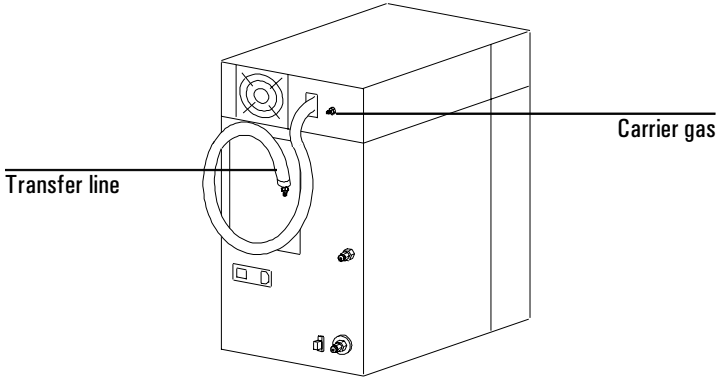
2. Remove the nut and Vespel/graphite ferrule from the carrier line. Keep the parts for later use.



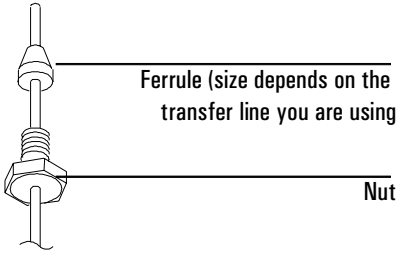
3. Slide a 1/16-inch Swagelok nut and then the Vespel/graphite ferrule from step 2 onto the end of the carrier line.



4. Connect the carrier line to the gas supply port labeled “Carrier Gas” on the back of the P&T concentrator using a 5/16-inch wrench. Tighten the nut 1/4-turn past finger tight. Do not overtighten. If the nut leaks, tighten an additional 1/8-turn until it seals.

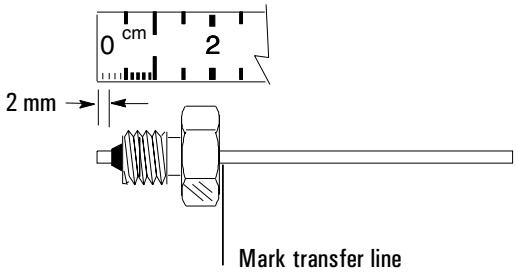


5. Slide the 1/16-inch male nut from step 2 and an appropriate Vespel/graphite ferrule onto the end of the P&T transfer line.

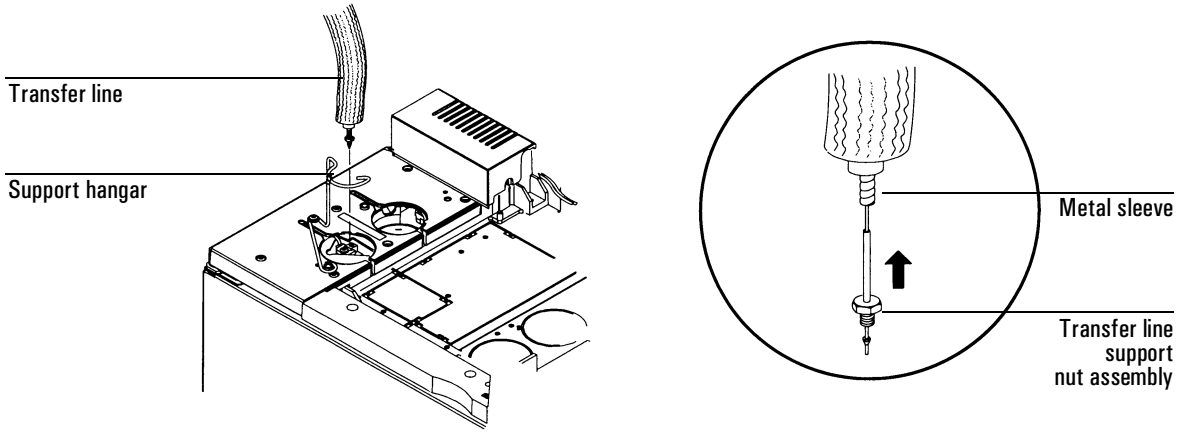


6. If you are using a *nickel-plated* transfer line, proceed to step 8. If you are using a *fused-silica transfer* line, prepare the end of the fused silica line.

7. Position the transfer line so that 2 mm of tubing is exposed in front of the ferrule, and mark the transfer line with typewriter correction fluid at a point even with the nut.

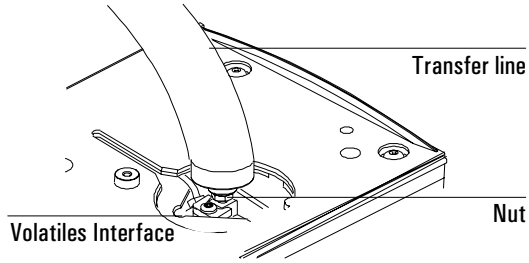


8. To connect the transfer line to the volatiles interface, first install the transfer line support nut assembly up and inside the metal sleeve of the heated transfer line assembly.



Then, connect the transfer line to the volatiles interface by finger tightening the 1/16-inch male nut while adjusting the transfer line's position so that the correction fluid mark stays aligned with the nut. Using a 1/4-inch wrench,

tighten the nut 1/4-turn past finger tight. Do not overtighten. If the fitting leaks, tighten an additional 1/8-turn until it seals.



9. After the column is installed at both the interface and the detector, establish a flow of carrier gas through the interface and maintain it for 10 to 15 minutes. Check for leaks. Heat the interface to operating temperatures and retighten the fittings, if necessary.

19 NonEPC Inlets

Purged packed inlet

Split/splitless inlet—split mode

Split/splitless inlet—splitless mode

Configuration

Procedure: Configuring a nonEPC inlet

Inlet control tables

Column control tables

Procedure: Setting carrier flow for the purged packed inlet

Procedure: Setting flows for the split mode inlet

Procedure: Setting flows for the splitless mode

NonEPC Inlets

Controls for these inlets are located on a pneumatics module attached to the left side of the GC.

Purged packed inlet

The only adjustment for this inlet is the carrier gas flow through the column. Septum purge flow is set automatically based on the source gas pressure. It can be measured at a vent on the front panel.

Split/splitless inlet—split mode

The carrier gas divides between the column and the split vent depending on their relative flow resistances. A small amount of carrier gas sweeps the lower side of the septum and exits through the septum purge control and vent.

Split/splitless inlet—splitless mode

In a splitless injection, a valve is actuated by [Prep Run] that prevents carrier gas from exiting the bottom of the inlet liner. Total flow does not change, but most of it exits through the septum purge line. All carrier gas that passes through the liner goes to the column—the sample is not split.

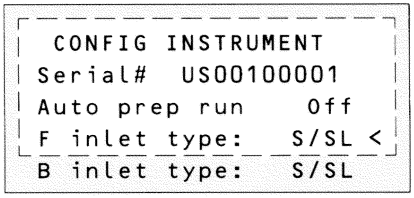
At purge time, the valve switches to sweep out residual vapor in the inlet. The system is now in the split configuration, with the purge flow and residual vapor—mostly solvent—exiting through the split vent.

Configuration

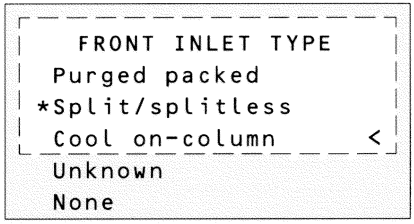
The GC is aware that a nonEPC inlet is present—it looks for the heater/sensor connections—but does not know what kind. You must supply this information through configuration.

Procedure: Configuring a nonEPC inlet

1. Press [Config], select Instrument, and [Enter].



2. Select the inlet and press [Mode/Type].



3. Select a type and [Enter].

4.Press [Config][Front Inlet] (or [Back Inlet].

5. Press [Mode/Type], select a gas, and [Enter].

Inlet control tables

The inlet control tables for nonEPC inlets are similar to those for the EPC versions except that flow and pressure settings are absent.

Purged packed inlet

```
    FRONT INLET (He)
Temp        150    150 <
```

Split/splitless inlet in split mode

```
    FRONT INLET (He)
Mode:            Split <
Temp        150    150
```

Split/splitless inlet in splitless mode

```
    FRONT INLET (He)
Mode:         Splitless <
Temp        150    150
Purge time        2.00
```

Figure 71 NonEPC inlet control tables

Column control tables

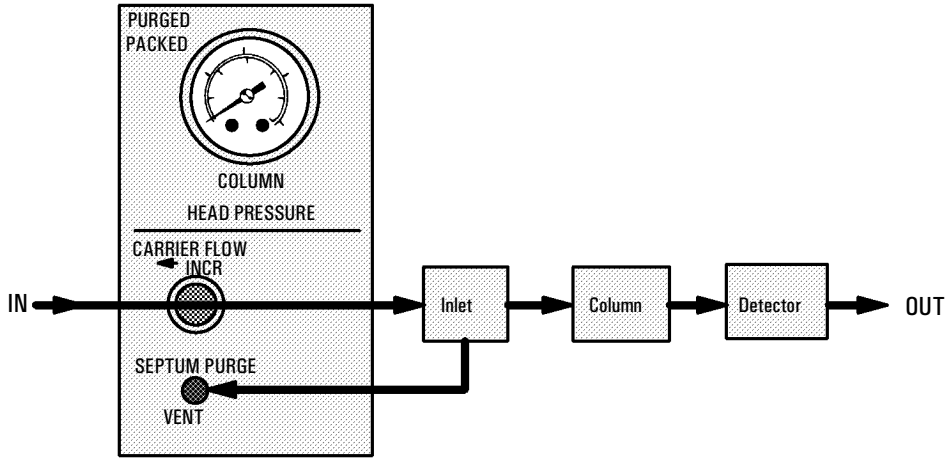
When a nonEPC split/splitless inlet is used with a defined column, the column control table becomes a calculator. Although you cannot control flows from the keyboard, you can determine the flows to be set manually.

```
Column 1 (He)
Dim    30.0 m   320 u
Pressure          0.0
Calc flow         0.0
Calc velocity       0
```

Enter a pressure. Flow and average linear velocity are calculated and displayed.

Procedure: Setting carrier flow for the purged packed inlet

The internal flow path in the instrument is:



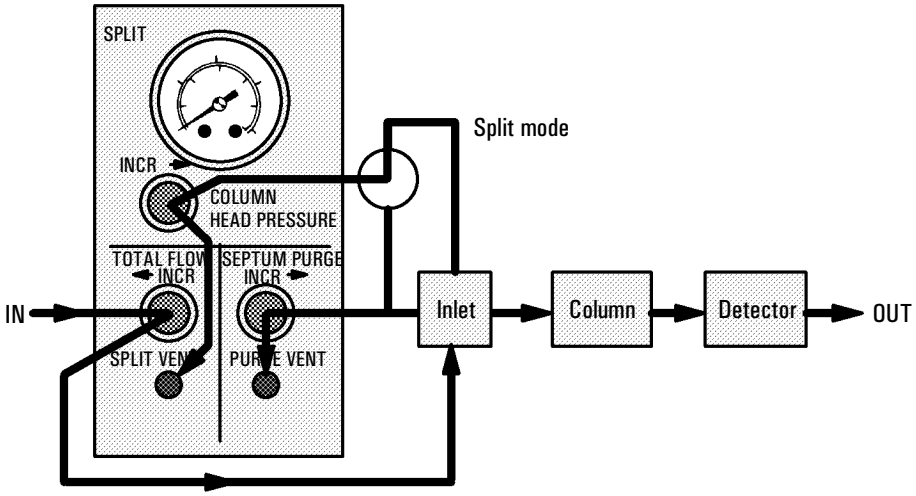
1. Locate the knob labeled CARRIER FLOW. Turn it *clockwise* as far as it will go. Do not force the knob; when it closes it comes to a slightly “soft” stop.
2. Open the carrier gas cylinder valve and set the delivery pressure of the two-stage regulator to 410 kPa (60 psi). If there is a local regulator in the carrier gas line, set the cylinder regulator to 550 kPa (80 psi) and the local regulator to 410 kPa (60 psi).

3. Attach a flow meter to the detector outlet. There should be no flow at this time. If there is, turn the detector gas flows off from the keyboard.
4. Turn the CARRIER FLOW knob in the ◄INCR direction to turn the carrier gas on. Adjust and measure to achieve the desired flow. If necessary, increase the source pressure.

The septum purge is set automatically.

Procedure: Setting flows for the split mode inlet

The internal flow path in the instrument is:

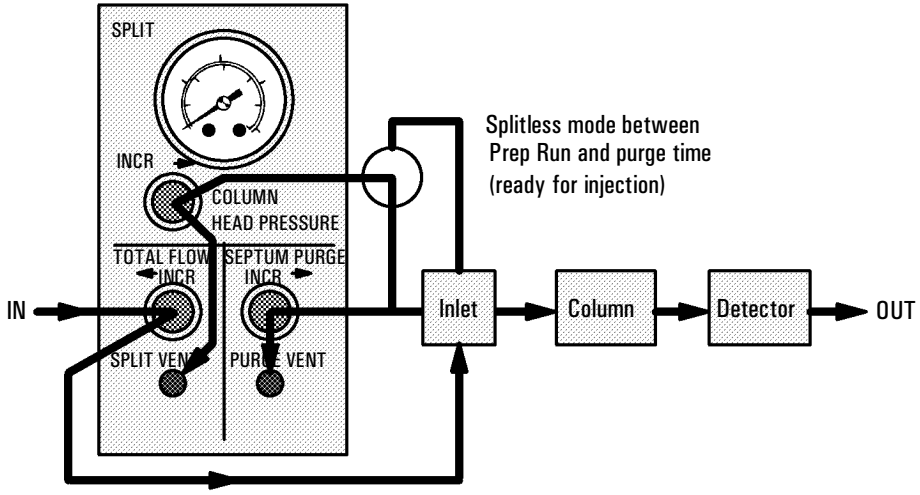


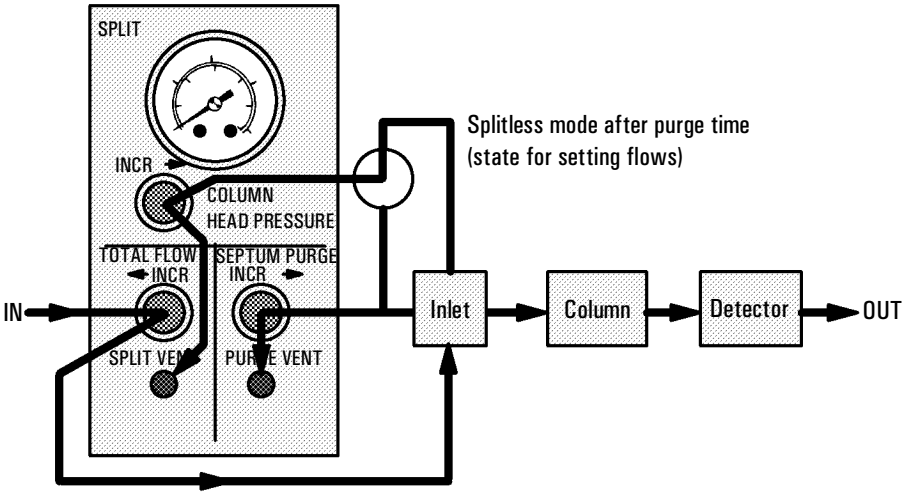
1. Locate the knob labeled TOTAL FLOW. Turn it *clockwise* as far as it will go. Do not force the knob; when it closes it comes to a slightly "soft" stop.
2. Locate the knob marked SEPTUM PURGE. Turn it *counterclockwise* to turn the flow off. There is no definite stop position; when the knob turns freely (does not seem to be touching anything inside), it is off.
3. Open the carrier gas cylinder valve and set the delivery pressure of the two-stage regulator to 410 kPa (60 psi). If there is a local regulator in the carrier gas line, set the cylinder regulator to 550 kPa (80 psi) and the local regulator to 410 kPa (60 psi). If you are using small-bore capillary columns, you may have to use higher pressures.

4. Attach a flow meter to the detector outlet. There should be no flow at this time. If there is, turn the detector gas controls off from the keyboard.
5. Turn the TOTAL FLOW knob in the ◄INCR direction to turn the carrier gas flow on.
6. Turn the COLUMN HEAD PRESSURE knob in the INCR► direction. Adjust and measure to achieve the desired column flow. If you cannot, increase TOTAL FLOW until you can. Use TOTAL FLOW for coarse and COLUMN HEAD PRESSURE for fine adjustment.
7. Move the flow meter to the SPLIT VENT. Measure and adjust TOTAL FLOW to achieve the desired split flow. If necessary, increase the source pressure.
8. Move the flow meter to the PURGE VENT. Turn the SEPTUM PURGE knob in the INCR► direction to achieve the desired septum purge flow.
9. Repeat steps 6, 7, and 8 until all flows are correct.

Procedure: Setting flows for the splitless mode

The internal flow paths in the instrument are:





1. Locate the knob labeled TOTAL FLOW. Turn it *clockwise* as far as it will go. Do not force the knob; when it closes it comes to a slightly "soft" stop.

2. Locate the knob marked SEPTUM PURGE. Turn it *counterclockwise* to turn the flow off. There is no definite stop position; when the knob turns freely (does not seem to be touching anything inside), it is off.

3. Open the carrier gas cylinder valve and set the delivery pressure of the two-stage regulator to 410 kPa (60 psi). If there is a local regulator in the carrier gas line, set the cylinder regulator to 550 kPa (80 psi) and the local regulator to 410 kPa (60 psi). If you are using small-bore capillary columns, you may have to use higher pressures.

4. Attach a flow meter to the detector outlet. There should be no flow at this time. If there is, turn the detector gas controls off from the keyboard.

5. Turn the TOTAL FLOW knob in the ◄INCR direction to turn the carrier gas flow on.

6. Turn the COLUMN HEAD PRESSURE knob in the INCR► direction. Adjust and measure to achieve the desired column flow. If you cannot, increase TOTAL FLOW until you can. Use TOTAL FLOW for coarse and COLUMN HEAD PRESSURE for fine adjustment.

7. Move the flow meter to the SPLIT/SPLITLESS INLET VENT. Measure and adjust TOTAL FLOW to achieve the desired split flow. If necessary, increase the source pressure.
8. Move the flow meter to the SEPTUM PURGE VENT. Turn the SEPTUM PURGE knob in the INCR► direction to achieve the desired septum purge flow.
9. Repeat steps 6, 7, and 8 until all flows are correct.

20 The Pneumatics Control Module

Using a Pneumatics Control Module

Operating the PCM

With an inlet

With a valve or other device

The control tables

Packed column or column not defined

Defined capillary columns

Procedure: Using packed and undefined capillary columns

Procedure: Using defined capillary columns

Maintaining a PCM

Procedure: Leak testing the gas plumbing

The Pneumatics Control Module

Using a Pneumatics Control Module

The Pneumatics Control Module (PCM) provides one channel of flow or pressure control, replacing the standard electronic flow control module (ECM) for that channel. It does not need to be connected to any particular type of inlet.

The PCM can control gas flows and pressures for a number of applications including:

- Non-Agilent standard inlets.
- Any valve application where no inlet is required. For example, the PCM can provide flow or pressure to a column connected to a gas sampling valve. Other valving applications may involve providing auxiliary gas flow, especially when using packed columns.
- Sample preparation devices. The Agilent Headspace Sampler and the Agilent Purge and Trap often require a controlled source of purge gas.
- Catalyst tubes or other conversion devices, such as the nickel catalyst tube. These devices often require a controlled source of makeup or reagent gas.

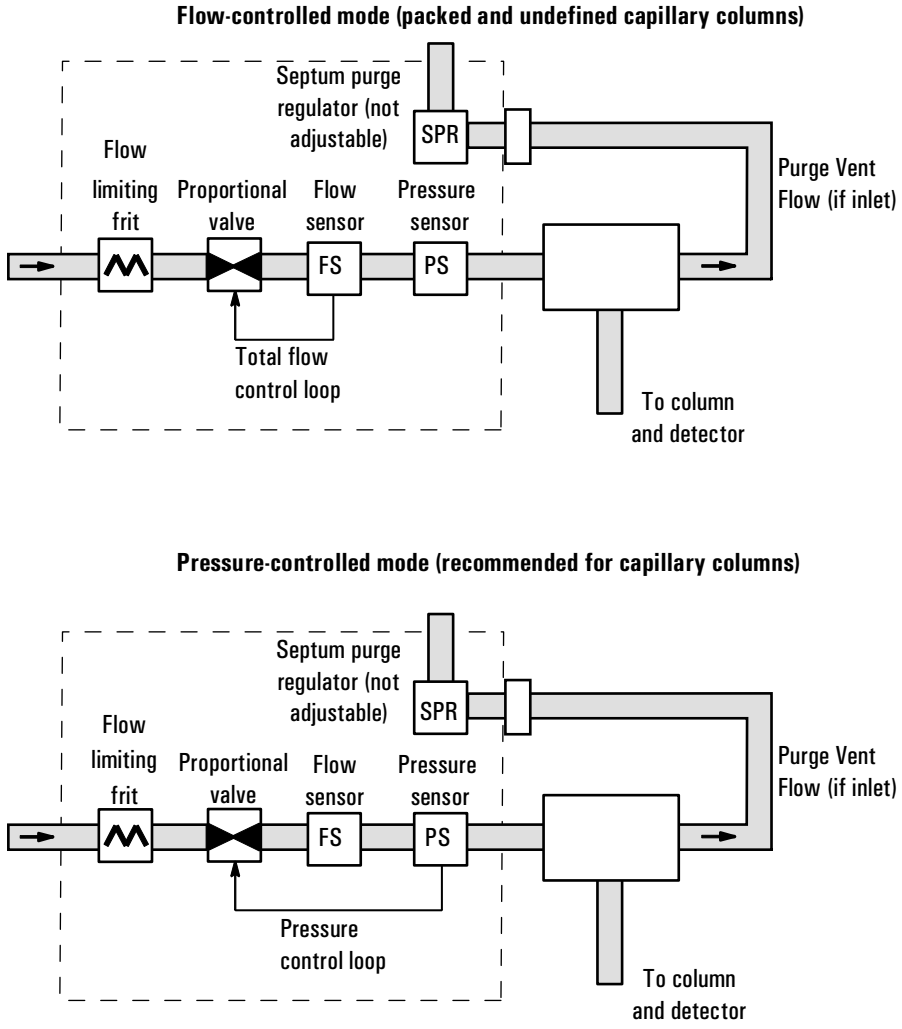


Figure 72 Pneumatics control module

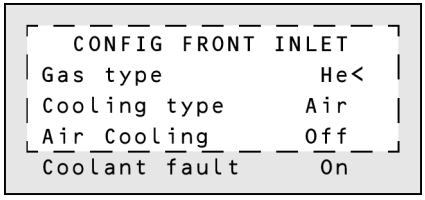
Operating the PCM

With an inlet

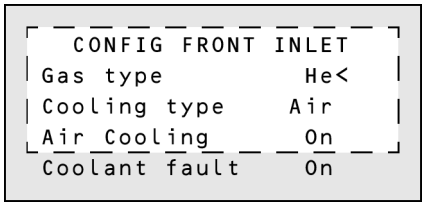
Some inlet types use compressed air cooling to reduce the thermal cycle time. The compressed air cooling must be configured before use.

To configure compressed air cooling:

1. Press the [Config] key followed by the [Front Inlet] key. Your display should be similar to the one below.



2. Scroll down to the line that displays `Air Cooling` and press the [ON] key. Your display should now look similar to the one below.



   When `Air Cooling` is On, power is available at the back valve connector to drive the compressed air actuator.

3. Your inlet is now configured for air cooling and ready for use.

With a valve or other device

When using the PCM with a valve, the PCM is often connected in series with the valve (or device) and column, providing regulated gas flow through the valve (or device) and onto the column.

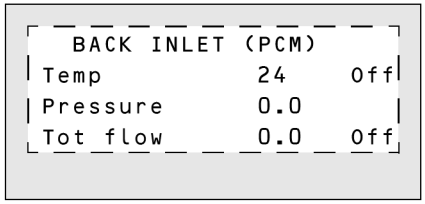
The control tables

The PCM can control either the flow to the inlet/valve/device or the pressure applied to a column attached to it. The column configuration uniquely determines whether the PCM delivers pressure control or flow control. If a capillary column is used and the column is defined, the inlet is pressure-

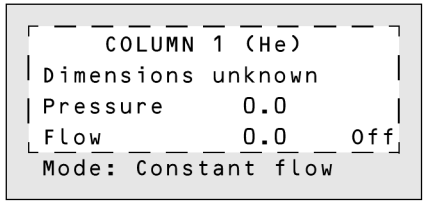
controlled. If the column is not defined (packed columns and undefined capillary columns), the inlet is flow-controlled.

For more details about the procedures for configuring columns, setting pressures, etc., see "Flow and Pressure Control".

Packed column or column not defined



Inlet



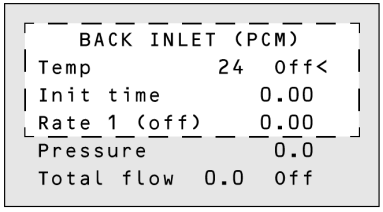
Column

`Temp` The setpoint and actual temperature values are shown if a heated inlet/device is installed.

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

`Tot flow` Enter your setpoint here; the actual value is displayed.

Defined capillary columns



Column defined

`Temp` The setpoint and actual temperature values are shown if a heated inlet/device is installed.

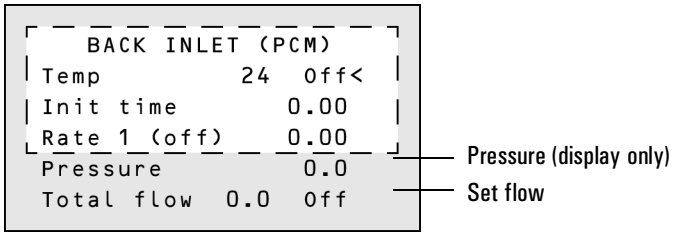
`Pressure` Inlet is pressure controlled. Enter your setpoint here (in psi, bar, or kPa) and the actual value is displayed.

`Tot flow` The actual total flow to the inlet. This is a reported value, not a setpoint.

Procedure: Using packed and undefined capillary columns

If the column is not defined, only the flow-controlled modes are available.

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. If an inlet is installed, press [Front Inlet] or [Back Inlet] and enter a temperature.



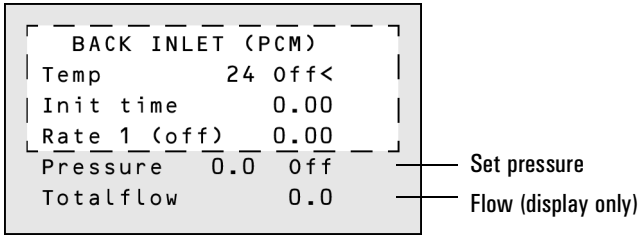
Column undefined

3. Inject the sample (or toggle the valve).

Procedure: Using defined capillary columns

The following procedure assumes that you have already set the flow or pressure.

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. If an inlet is installed, press [Front Inlet] or [Back Inlet] and enter a temperature.



Column Defined

3. Inject the sample (or toggle the valve).

Maintaining a PCM

Procedure: Leak testing the gas plumbing

Leaks in the gas plumbing system can affect chromatographic results dramatically.

Liquid leak detectors are not recommended, especially in areas where cleanliness is very important.

If you do use leak detection fluid, immediately rinse the fluid off to remove the soapy film.

WARNING To avoid a potential shock hazard when using liquid detection fluid, turn the GC off and disconnect the main power cord. Be careful not to spill leak solution on electrical leads, especially the detector heater leads.

Materials needed:

- Electronic leak detector (recommended) or liquid leak detection fluid. If you use leak detection fluid, wipe off excess fluid when you have completed the test.
- Two 7/16-inch wrenches

1. Using the leak detector, check each connection you have made for leaks. Check the connections leading to and from the PCM.
2. Correct leaks by tightening the connections. Retest the connections; continue tightening until all connections are leak-free.

If the PCM (rather than its connections) is leaking, contact Agilent service.

21 Using Detectors

Using hydrogen
Procedure: Setting up detector control tables

Makeup gas flow
Makeup gas
Procedure: Defining the makeup gas
Procedure: Changing makeup gas flow mode

Maximum flow rates

[Det Control] shortcut key

Using Detectors

The 6890 Series gas chromatograph (the GC) has several detector systems available. Others will be added in the future. See your Agilent sales representative for the latest information.

Name	Sensitivity	Responds to	Comments
Thermal conductivity, TCD	Medium	Everything except the carrier gas	The "Universal Detector" for everything
Flame ionization, FID	High	Almost all organic compounds	The "Universal Detector" for organics
Micro-electron capture, μ-ECD	Very high	Limited range of compounds, mostly halocarbons	Used for trace level pesticide and herbicide analysis
Nitrogen-phosphorus, NPD	Very high	Compounds with nitrogen or phosphorus	Used in pharmaceutical and environmental analysis
Flame photometric, FPD	High	Compounds with sulfur or phosphorus	Used in environmental and bioscience analysis

Using hydrogen

WARNING When using hydrogen (H_2) as the carrier gas or fuel gas, be aware that hydrogen (H_2) gas can flow into the oven and create an explosion hazard. Therefore, be sure that the supply is off until all connections are made, and ensure that the inlet and detector column fittings are either connected to a column or capped at all times when hydrogen (H_2) gas is supplied to the instrument.

WARNING Hydrogen (H_2) is flammable. Leaks, when confined in an enclosed space, may create a fire or explosion hazard. In any application using hydrogen (H_2), leak test all connections, lines, and valves before operating the instrument. Always turn off the hydrogen (H_2) supply at its source before working on the instrument.

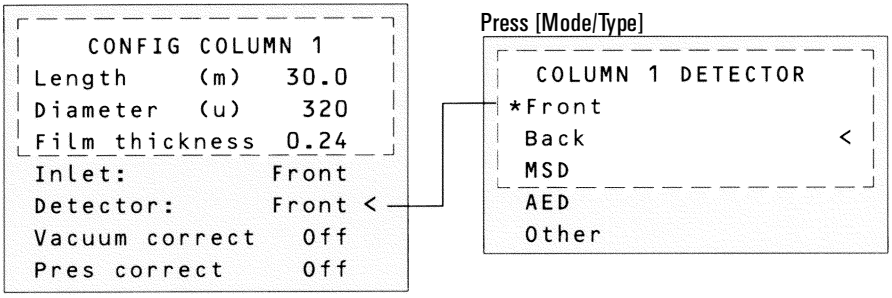
Procedure: Setting up detector control tables

You must be familiar with this set of control tables to operate your detector. Follow these three steps when setting up all types of detectors.

1. Check your column configuration. (This is normally done when you set up your inlet, but it does not hurt to check this information again.)

   - You must tell the instrument which detector your column is connected to, front or back. If you have only one detector, it is best to have only one column configured to it—unless you actually do have two columns attached to that detector.
   - If you are using a capillary column, you must enter the column length and diameter if you want to have a choice of makeup gas flow modes. This is called *column defined*. If you do not enter these values, it is called *column not defined*, and your control choices are limited.

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



2. Scroll to `Detector`: Press [Front] or [Back] or Press [Mode/Type] and choose `Front` or `Back`
3. Enter column dimensions, if necessary.

2. Check your detector configuration (makeup gas type).

   The main reason for doing this is to verify that the makeup gas entered (or makeup and anode gas on the ECD, reference and makeup on the TCD) is the same as the gas plumbed to your detector.

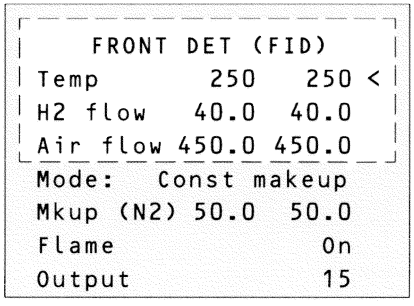
   This is important because:

   - When the actual and configured gas types are different for an EPC detector, the calculated flow rate is not correct and the flow rate stability is affected.
   - The electronics for some detectors change depending on the gas type configuration. The detector does not operate properly when the actual and configured gas types are different.
   - Good laboratory practice. Keep a record of the gas used.

   Most of the detectors have other configurable items. These will be described later in this section.

3. Set up your detector control table. The following is a brief description of each line item for the FID. Flow setpoints (right number) and actual values (left number) are displayed.

Press [Front Det] or [Back Det].
(column not defined)



`FRONT DET (FID)` The title indicates the detector position and the type of detector installed.

`Temp` This is where you set the temperature - the control setpoint (right number) and actual (left number) values.

`H2 flow, Air flow` These are the detector gases for the FID.

`Mkup (N2)` This is where you set your makeup gas flows. The gas type is displayed in parentheses. The lines of the display vary depending on your instrument and the way you have configured it.

`Flame` This is the on/off control for the FID—also called the Detector Control line. Each detector has its own type of on/off control.

`Output` This is the actual detector output value. You cannot enter a setpoint here.

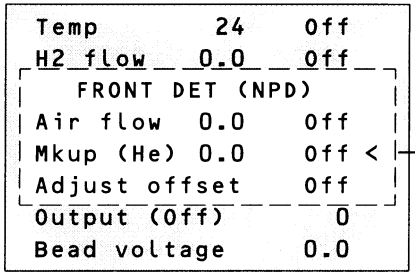
Makeup gas flow

Makeup gas enters the detector close to the end of the column. Its purpose is to speed the peaks through the detector—especially with capillary columns—so that the peak separation achieved by the column is not lost through remixing in the detector.

Makeup gas

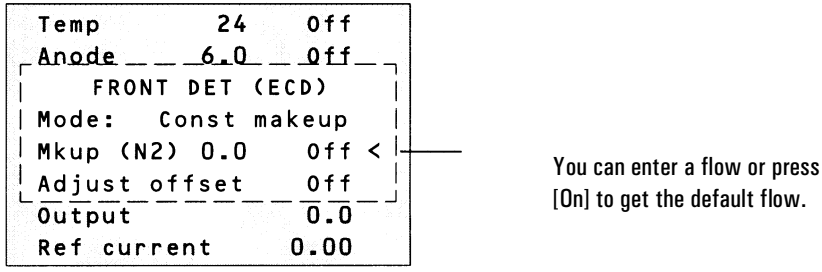
The makeup gas line of your detector control table changes depending on your instrument configuration.

If you have an inlet with the *column not defined*, the makeup flow is constant. The control table for the detector looks like this:

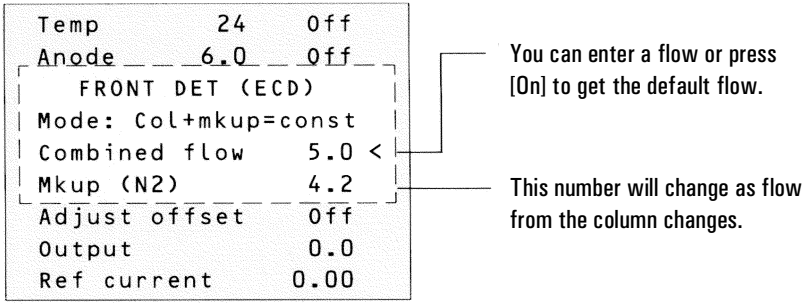


If you are operating with *column defined*, you have a choice of two makeup gas modes.

The `Const makeup` mode provides a constant flow of makeup gas to the detector. If you choose it, your control table looks like this:

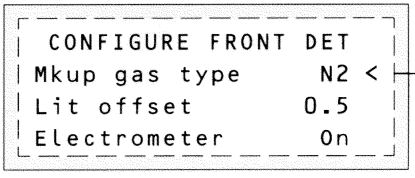


The `Col+mkup=const` mode provides a variable flow of makeup gas to the detector. As column flow increases or decreases, the makeup flow changes to provide a constant combined flow to the detector. If you choose this option, enter a value under `Combined flow.` The `Combined flow` line always displays the same value, while the `Mkup` line of the control table changes as the actual makeup flow changes.

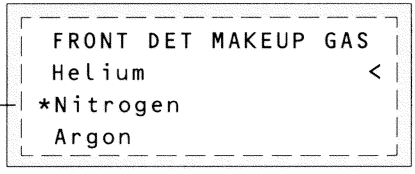


Procedure: Defining the makeup gas

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



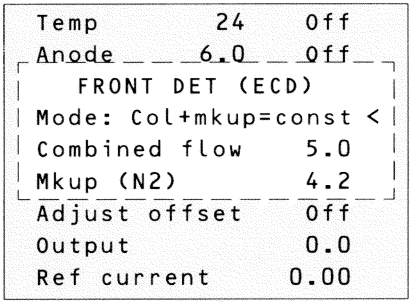
2. Scroll to `Mkup gas type` and press [Mode/Type].



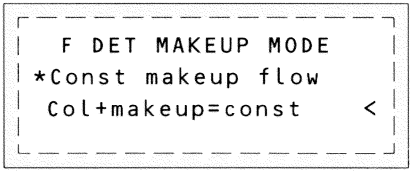
3. Scroll to the correct gas and press [Enter].

Procedure: Changing makeup gas flow mode

1. Scroll to `Mode:`



2. Press [Mode/Type].



3. Choose a flow mode and press [Enter].

Maximum flow rates

The maximum flow rates that can be set with detectors are:

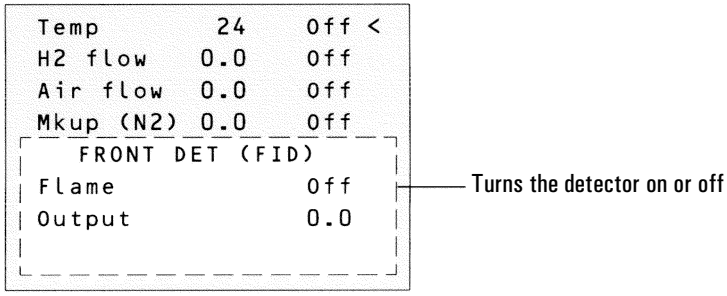
Detector and gas	Maximum flow rate, mL/min
Flame ionization	
Hydrogen	100
Air	800
Makeup (nitrogen, helium, argon)	100
Thermal conductivity	
Nitrogen	reference 100; makeup 10
Helium	reference 100; makeup 12
Hydrogen	reference 100; makeup 18
Argon	reference 100; makeup 10
Micro-electron capture	
Nitrogen	anode purge 12; makeup 200
Argon/methane	anode purge 12; makeup 200
Nitrogen-phosphorus	
Hydrogen	30
Air	200
Makeup (nitrogen, helium, argon)	100
Flame photometric	
Hydrogen	250
Air	200
Makeup (nitrogen, helium, argon)	130

[Det Control] shortcut key

This is another way to open a Detector control table. Press [Front Det] [Det Control] or [Back Det] [Det Control] to open a Detector control table. If you have only one detector, [Det Control] opens that table.

When you use [Det Control], your table opens at the On/Off control for your detector—FID and FPD, `Flame`, TCD `Filament`, NPD, `Adjust offset`.

Press [Det Control]



22 The Flame Ionization Detector

General Information

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- Detector shutdown

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Procedure: Replacing the FID ignition wire

The Nickel Catalyst Tube

Gas flows

Temperature

Repacking the catalyst

The Flame Ionization Detector

General Information

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

FID pneumatics

Figure 73 illustrates the pneumatics design for the FID.

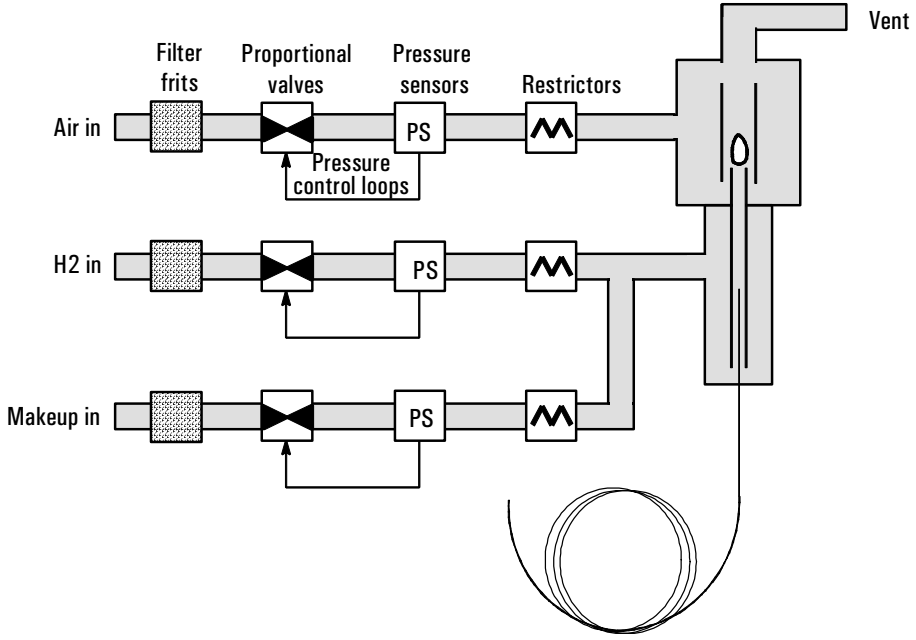


Figure 73 Schematic of a flame ionization detector

Special considerations

Conditions that prevent the detector from operating

- Temperature set below 150°C
- Air or hydrogen flow set at `Off` or set at 0.0
- Ignition failure

Detector shutdown

If a critical detector gas is shut down due to a pneumatics or ignition failure, your detector shuts down. This turns off everything except the detector temperature and makeup gas flow.

Jets

There are two types of FID available. The *capillary optimized* FID is only used with capillary columns, and the *adaptable* FID fits packed columns and can be adapted to fit capillary columns.

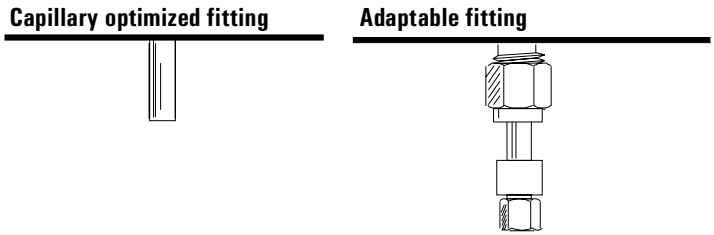


Table 59 Jets for the Capillary-Optimized FID

Jet type	Part no.	Jet tip id
Capillary	G1531-80560	0.29 mm (0.011-inch)
High-temperature *(use with simulated distillation)*	G1531-80620	0.47 mm (0.018-inch)

Table 60 Jets for the Adaptable FID

Jet type	Part no.	Jet tip id
Capillary	19244-80560	0.29 mm (0.011-inch)
Packed	18710-20119	0.47 mm (0.018-inch)
Packed wide-bore *(use with high-bleed applications)*	18789-80070	0.030-inch
High-temperature *(use with simulated distillation)*	19244-80620	0.47 mm (0.018-inch)

Your detector is shipped with a capillary column jet. If you are doing simulated distillation or high-temperature runs, you must change the jet. Instructions appear in "Replacing or cleaning the jet".

Automatic reignition—Lit offset

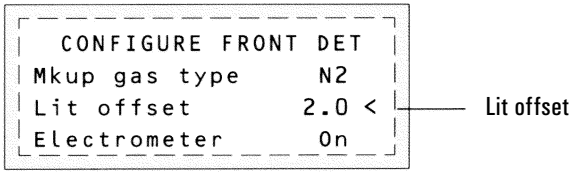
`Lit offset` is the expected difference between the FID output with the flame lit and the output with the flame off. If the output falls below this value, the FID will attempt to reignite twice. If the output does not increase by at least this value, the detector shuts down all functions except temperature and makeup gas flow.

The default setting for `Lit offset` is 2.0 picoamps. This is a good working value for all but very clean gases and systems. You may want to change this setpoint if:

- Your detector is attempting to reignite when the flame is still on, thus producing a shutdown.
- Your detector is not trying to reignite when the flame is out.

Procedure: Changing the auto-reignite setpoint

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



2. Scroll to `Lit offset` and enter a number.
The default is 2.0 pA.
Enter 0 to disable the automatic reignite function.
The setpoint range is 0 to 99.9 pA.

Electrometer

The Configure Detector control table contains an `On/Off` setpoint for the `Electrometer`. You do not need to turn the electrometer on and off when operating your FID. The only time you need to turn off the electrometer is when cleaning the detector.

Caution Do not turn off the electrometer during a run. It will cancel detector `Output`.

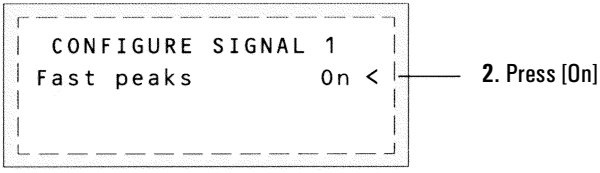
Data rates

Analog output for the FID can be presented at either of two speeds. The faster speed allows minimum peak widths of 0.004 minutes, while the standard speed allows peak widths of 0.01 minutes.

Procedure: Using fast peaks

If you are using the *fast peaks* feature, your integrator must be fast enough to process the data coming from the GC. It is recommended that your integrator bandwidth be at least 15 Hz. To use fast peaks:

1. Press [Config][Signal 1] or [Config][Signal 2]



Digital output to the ChemStation is available at eleven speeds ranging from 0.1 Hz to 200 Hz, capable of handling peaks from 0.001 to 2 minutes wide. Consult "Signal Handling".

The fast peaks feature does not apply to digital output.

Operating the FID

Use the information in Table 61 when selecting temperatures and flows. Choose a minimum source pressure from Figure 74.

Table 61 Recommended Temperature and Flow Rates—FID

Gas	Flow range (mL/min)	Suggested flow (mL/min)
Carrier gas *(hydrogen, helium, nitrogen)*		
Packed columns	10 to 60	
Capillary columns	1 to 5	
Detector gases		
Hydrogen	24 to 60*	40
Air	200 to 600*	450
Column plus capillary makeup *Recommended: nitrogen* *Alternate: helium*	10 to 60	50
Detector temperature		
< 150° C, flame will not light, prevents condensation damage Detector temperature should be approximately 20°C greater than highest oven ramp temperature depending on the column type.		
Lit offset [Config][Front Det] or [Config][Back Det] If the detector output (when the flame is on) minus the detector output (when the flame is off) falls below this value, the FID attempts to reignite twice. If output does not increase by at least this value, the detector shuts down. 2.0 pA is the recommended setting. 0.0 pA disables the autoreignite function.		
*The hydrogen-to-air ratio should be between 8% and 12% to keep the flame lit.		

Gas pressures

Choose a flow, find a pressure. Set source pressure 10 psi (70 kPa) higher.

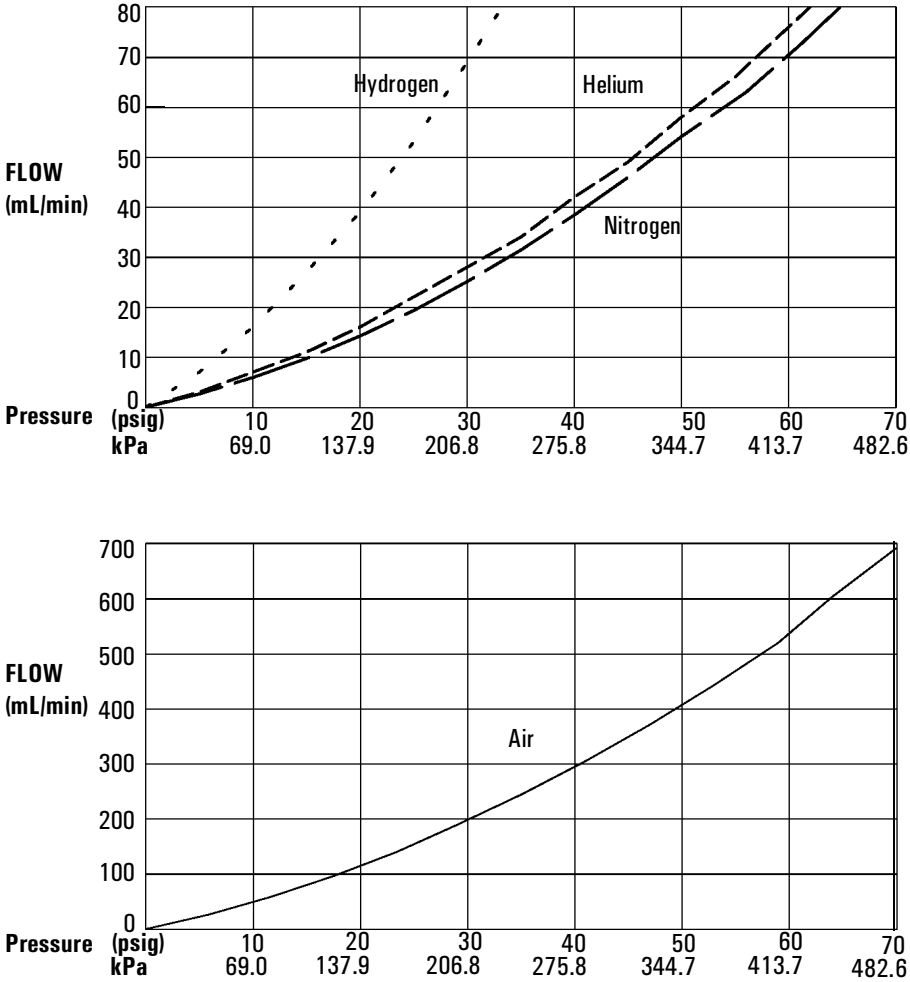
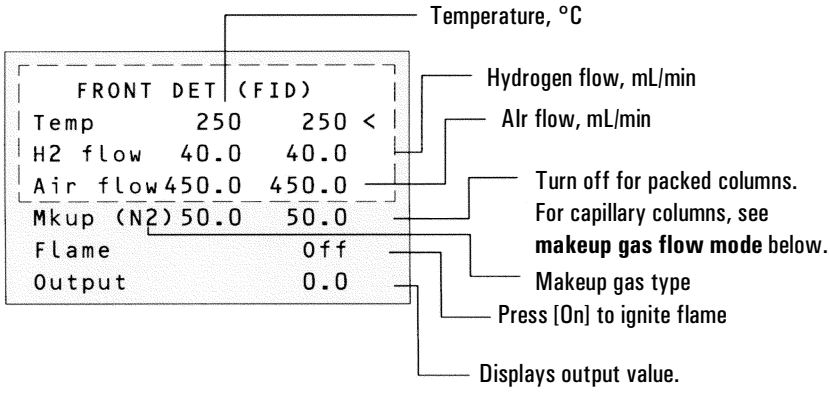


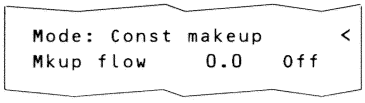
Figure 74 Typical pressure/flow relationships for FID gases
(at 25°C and 1 atmosphere of pressure)

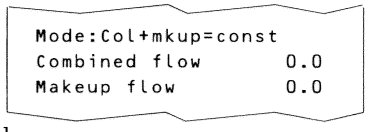
Operating with EPC

Press [Front Det] or [Back Det].

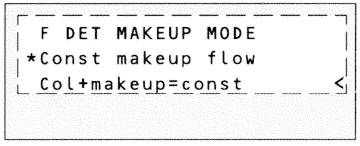


Makeup gas flow mode:
If column dimensions are specified, the control table will also include one of these:

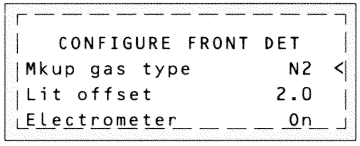




To **change makeup mode,** scroll to Mode: and press [Mode/Type]. Make a selection and enter the appropriate flow values.



To view **makeup gas** or change **Lit offset,** press [Config][Front Det] or [Config][Back Det]:



It is not necessary to turn the electrometer on or off unless you are performing a maintenance procedure.

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

Select the appropriate gas.

Figure 75 FID control table

Procedure: Using the FID

Verify that all detector gases are connected, a column is installed, the correct jet is installed, and the system is free of leaks. Check the oven temperature, inlet temperature, and column flow. Use Figure 75 as a guide when operating the FID.

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

1. Press [Front Det] or [Back Det] to open the FID control table.
2. Set the detector temperature. The temperature must be greater than 150°C for the flame to light.
3. Change the hydrogen flow rate, if desired, and press [Off].
4. Change the air flow rate, if desired, and press [Off].
5. If you are using ***packed columns***, turn off the makeup gas and proceed to Step 7.
6. If you are using ***capillary columns***:
   a. Verify that makeup gas type is the same as that plumbed to your instrument (next to `Mkup` line of control table). Change the gas type, if necessary.
   b. If your capillary column is *defined* and connected to an EPC inlet, choose a new flow mode, if desired, and set the makeup gas flow or combined flow.
   c. If your capillary column is *not defined* or connected to a nonEPC inlet, enter a makeup gas flow. Only constant flow is available in this case.
7. Scroll to `Flame` and press [On]. This turns on the air and hydrogen and initiates the ignition sequence. The signal typically increases to 5 to 20 pA after ignition. Verify that the flame is lit by holding a cold, shiny surface, such as a mirror or chrome-plated wrench, over the collector exit. Steady condensation indicates that the flame is lit.

Short-cut procedure:
(assumes correct setpoints are stored)

1. Open detector control table.
2. Turn temperature On.
3. Turn makeup gas On, if needed.
4. Press [Det Control].
5. Press [On].

Checkout Conditions and Chromatogram

This section contains a typical examples of a test sample chromatogram. It may be used as a general guide to instrument performance.

Note that injection volumes listed with operating conditions do not necessarily indicate total absolute volume injected. Volume given is simply the graduation (plunger position) read from a standard 10 µL syringe. For a heated inlet, actual sample volume injected will also include an additional 0.4-0.7 µL, the volume of sample volatilized from inside the syringe needle. For the dedicated, on-column inlet (unheated), the syringe plunger position more accurately reflects the true injected volume.

Also note that the following procedure and results are intended only to provide evidence of a properly functioning inlet and/or detector system; they are not necessarily suitable to test a given system against its specification limits.

FID checkout conditions

Column and sample

Type	HP-5 30m x 0.32mm x 0.25μm PN 19091J-413
Sample	FID Checkout 18710-60170
Injection volume	1 μL

Inlet

Temperature	250° C Purged/Packed or Split/Splitless
	Oven Track Cool On-Column
	40°C PTV (see below)
Inlet pressure	25 psi (Constant pressure, helium)

Split/Splitless

Mode	Splitless
Purge flow	60 mL/min
Purge time	0.75 min

Inlet, continued

PTV	
Mode	Splitless
Inlet temperature	40°C
Initial time	0.1 min
Rate 1	720°C/min
Final temp 1	350°C
Final time 1	2 min
Rate 2	100°C/min
Final temp 2	250°C
Final time 2	0 min
Inlet pressure	25 psi (Constant pressure)
Purge time	0.75 min
Purge flow	60 mL/min

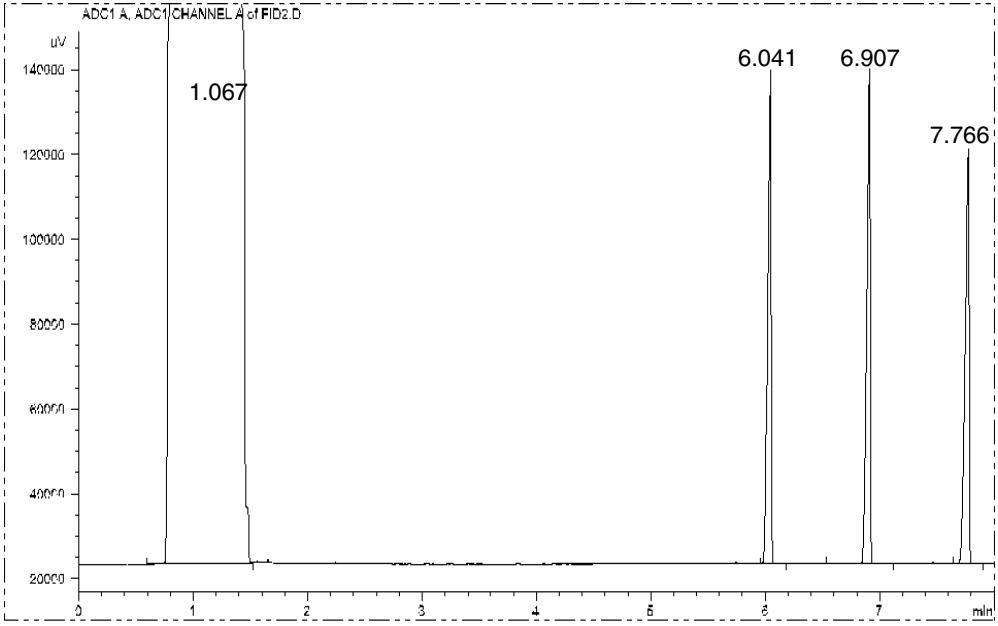
Detector

Temperature	300° C
H_2 flow	30 mL/min
Air flow	400 mL/min
Makeup flow (N_2)	25 mL/min
Offset	Should be < 20 pA

Oven

Initial temp	40° C
Initial time	0 min
Rate 1	25° C/min
Final temp	90° C
Final time	0 min
Rate 2	15° C/min
Final temp	170° C
Final time	2 min

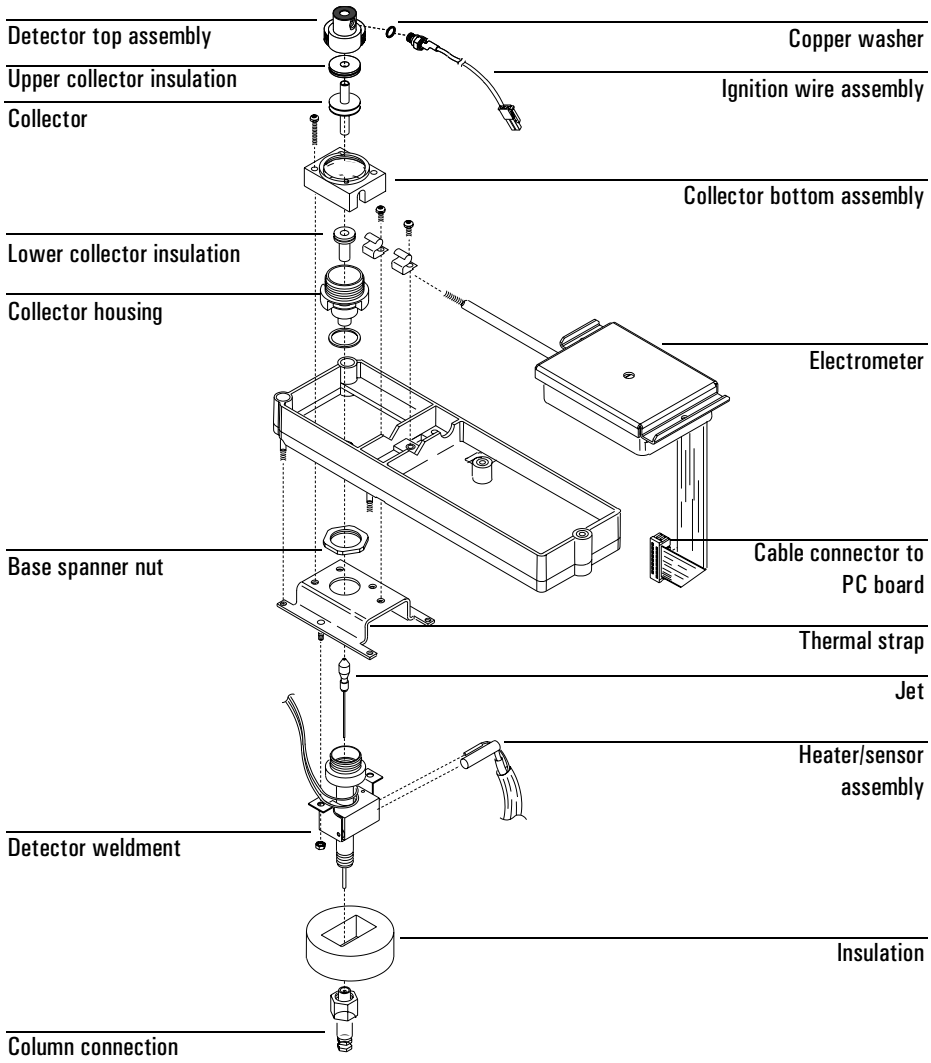
Typical FID checkout chromatogram



Your retention times will differ, but the peaks should be symmetric as in this example.

Maintaining a Flame Ionization Detector

WARNING Flame ionization detectors use hydrogen gas as fuel. If hydrogen flow is on and no column is connected to the detector inlet fitting, hydrogen gas can flow into the oven and create an explosion hazard. Detector fittings must have either a column or a cap connected at all times.



Correcting FID hardware problems

The flame goes out or will not light

- Check the column flow rate. It may be too high. Decrease the flow rate or pressure. Switch to a more restrictive column (longer or with a smaller id). If you must use a large id column, turn off the carrier flow long enough to allow the FID to light. Check for partially or completely plugged jet.
- Check that the right type of jet is installed for the column you are using. Jet types are listed on page 517.
- Injecting large volumes of aromatic solvent can cause the flame to go out. Switch to a nonaromatic solvent.
- The lit offset value may be too low or too high. Adjust the value.

WARNING Flame ionization detectors use hydrogen gas as fuel. If hydrogen flow is on and no column is connected to the detector inlet fitting, hydrogen gas can flow into the oven and create an explosion hazard. Detector fittings must have either a column or a cap connected at all times.

Replacing or cleaning the jet

Jets require periodic cleaning or replacement. Even with normal use, deposits develop in the jet (usually white silica from column bleed or black, carbonaceous soot). These deposits reduce sensitivity and cause chromatographic noise and spikes. Although you can clean the jet, it is usually more practical to replace dirty jets with new ones. If you do clean the jet, be very careful not to damage it.

You may also need to change the jet when you change columns or analyses. For example, packed columns use different jets than capillary columns. You must install the proper jet *before* changing the column.

To change a jet, you must first remove the FID collector assembly. The procedure is divided into three parts: removing and inspecting the jet, cleaning the jet (optional), and installing the jet.

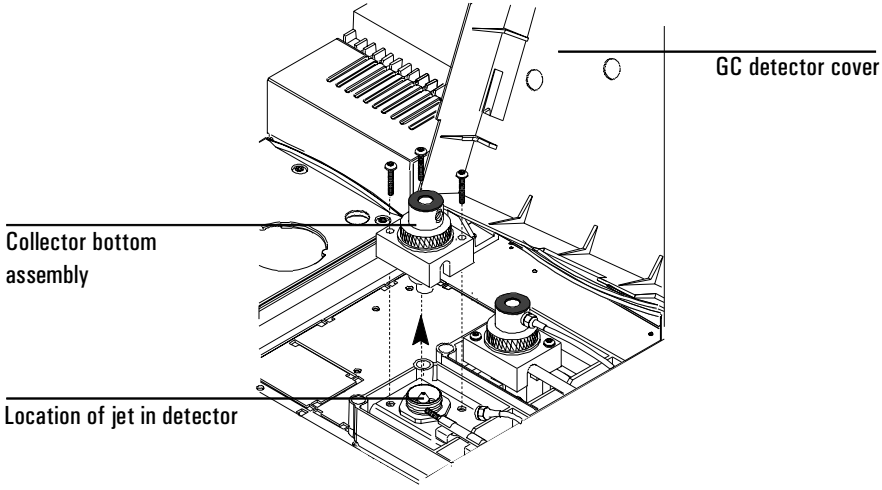
Procedure: Removing and inspecting the jet

Materials needed:

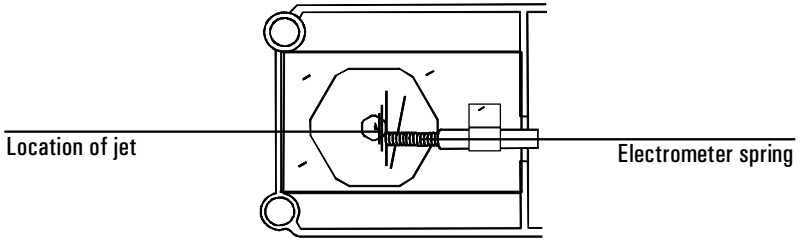
- Gloves to protect hands if detector is hot
- T-20 Torx screwdriver
- 1/4-inch nut driver
- Forceps (or tweezers)

1. Complete the following preliminary steps:
   - Cool the detector to room temperature.
   - When the detector is cool, turn it off and turn off the gases at the GC keyboard.
   - Turn off the electrometer; press [Config] [Front Det] or [Config] [Back Det] to access the control table.
   - Cool the inlet and then turn off the inlet gas.
   - Cool the oven, remove the column, and plug the column connection. See "Columns and Traps" .
   - Open the GC detector cover to access the FID.

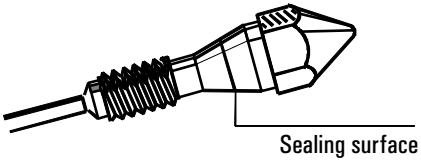
2. Put the gloves on if the detector is hot. Remove the three screws holding the collector bottom assembly in place. Lift off the assembly. The insulator can remain in the collector bottom.



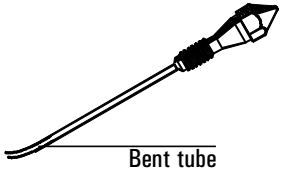
3. Using the nut driver, loosen the jet, and pull it straight out. You may need to use the forceps to grasp the jet.



4. Inspect the jet sealing surface for scratches. You should see a ring around the sealing surface; any other scratches, however, are unacceptable.



5. Inspect the jet tube to make sure it is not bent or crimped. Inspect the jet for contamination or pieces of broken column by holding it up to a light and looking through it. If no contamination is present, the tube will be clear.



Procedure: Cleaning the jet

It is often more convenient to replace dirty jets with new ones than to clean them, especially jets that have been badly contaminated.

If you choose to clean a jet, be careful when using a cleaning wire. Be sure not to scratch the jet internally, because doing so will ruin it. You may want to skip cleaning the jet with a wire and use the aqueous bath only.

Materials needed:

- Small ultrasonic cleaning bath
- Aqueous detergent
- GC-grade methanol in a Teflon wash bottle
- Flame detector cleaning kit (part no. 9301-0985)
- Dry, filtered, compressed air or nitrogen
- Forceps or tweezers

1. Run a cleaning wire through the top of the jet. Run it back and forth a few times until it moves smoothly. Be careful not to scratch the jet.

2. Aqueous cleaning procedure:

    a. Fill the ultrasonic cleaning bath with aqueous detergent and place the jet in the bath. Sonicate for 5 minutes.

    b. Use a jet reamer to clean the inside of the jet.

    c. Sonicate again for 5 minutes.
    From this point on, handle the parts only with forceps (or tweezers)!

    d. Remove the jet from the bath and rinse it thoroughly with hot tap water and then with a small amount of methanol.

    e. Blow the jet dry with a burst of compressed air or nitrogen and then place the jet on a paper towel to air dry.

Procedure: Installing the jet

Caution Do not over-tighten the jet! Over-tightening may permanently deform and damage the jet, the detector base, or both.

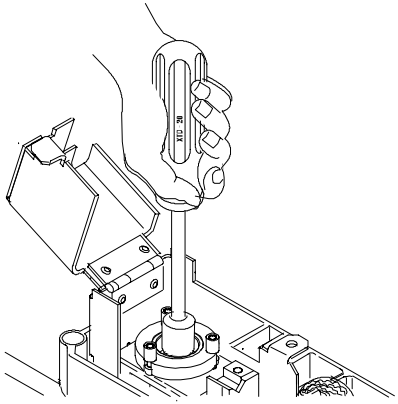
Caution Handle the clean or new jet only with forceps!

Materials needed:

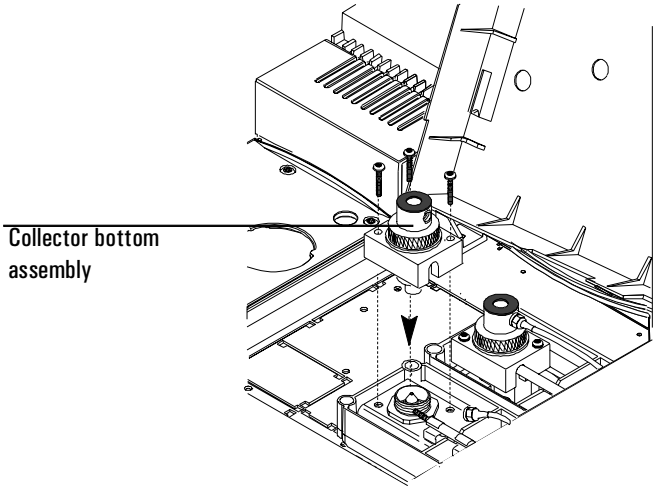
- Gloves to protect hands if detector is hot
- Forceps
- 1/4-inch hex driver
- T-20 Torx screwdriver

See page 517 for tables of jet types.

1. Insert the jet and tighten with the hex driver until it is snug.



2. Replace the collector assembly. Tighten the three screws securing the collector assembly.



3. Reattach the column to the detector. You can now restore normal operating conditions.

Cleaning the collector

The collector requires occasional cleaning to remove deposits (usually white silica from column bleed, or black, carbonaceous soot). Deposits reduce sensitivity and cause chromatographic noise and spikes.

The cleaning procedure presented here suggests you use an ultrasonic bath to clean the collector and other parts of the detector. However, if your collector is not too dirty, it may be sufficient to scrub it with a nylon brush and then use a burst of compressed air or nitrogen to blow stray particles away.

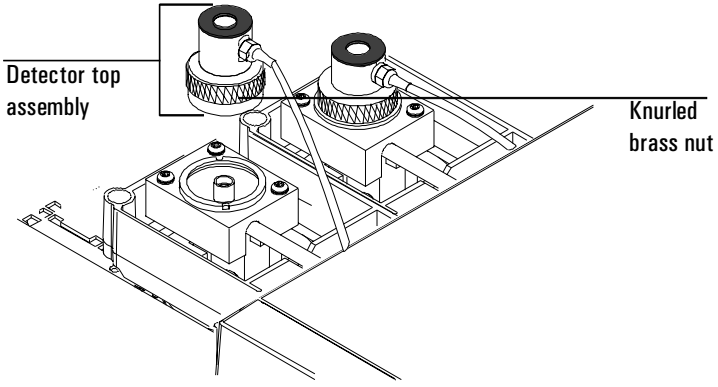
This procedure is divided into three steps: removing the collector, cleaning the collector, and reassembling the detector.

Procedure: Removing the collector

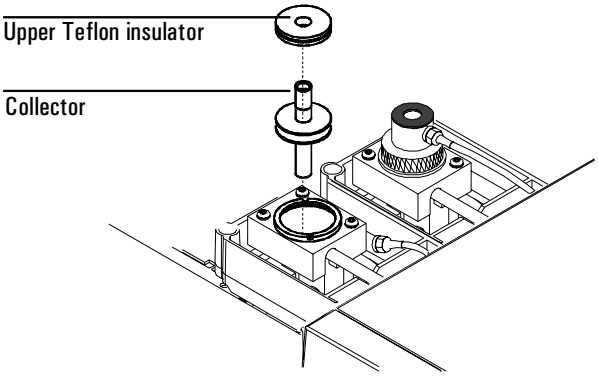
Materials needed:

- T-20 Torx screwdriver
- 1/4-inch nut driver
- Forceps or tweezers
- Gloves if the detector is hot

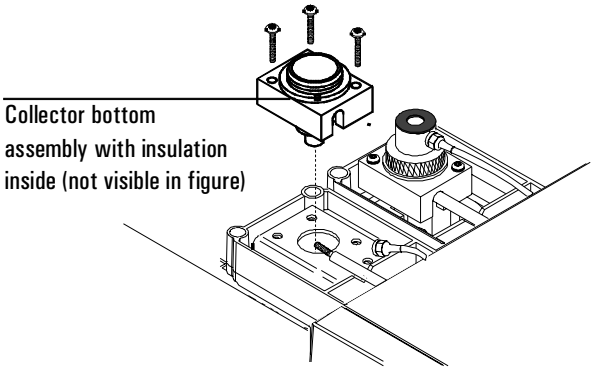
1. Complete the following preliminary steps:
   - Cool the detector to room temperature.
   - When the detector is cool, turn off the temperature zone and the gases at the GC keyboard.
   - Turn off the electrometer; the electrometer control is in the Config Det table. Press [Config] [Front Det] or [Config] [Back Det] to access the control table.
   - Open the GC detector cover to access the FID.
2. Put on the gloves if the detector is hot. Loosen the knurled brass nut. Lift the top assembly straight up. The upper Teflon insulator might stick to the bottom of the assembly. Remove the insulator.



3. Lift out the collector. The upper insulator may be attached to the collector. You may need to use the tweezers to grasp the collector.



4. Remove the three screws that hold the collector bottom assembly in place. Lift off the assembly. Remove the lower insulator from the bottom assembly. You may need to use the forceps to grab it.



Procedure: Cleaning the collector

Materials needed:

- Small ultrasonic cleaning bath
- Aqueous detergent
- GC-grade methanol in a Teflon wash bottle
- Flame detector cleaning kit (part no. 9301-0985)
- Dry, filtered, compressed air or nitrogen
- Forceps or tweezers

Cleaning procedure:

1. Fill the ultrasonic cleaning bath with aqueous detergent, and place the two insulators and the collector in the bath. Sonicate for 5 minutes.
2. Use the nylon brushes to clean each piece.
3. Sonicate again for 5 minutes.
   From this point on, handle the parts only with forceps or tweezers!
4. Remove the pieces from the bath and rinse them thoroughly with hot tap water and then with a small amount of methanol.
5. Place the pieces on a paper towel to air dry.

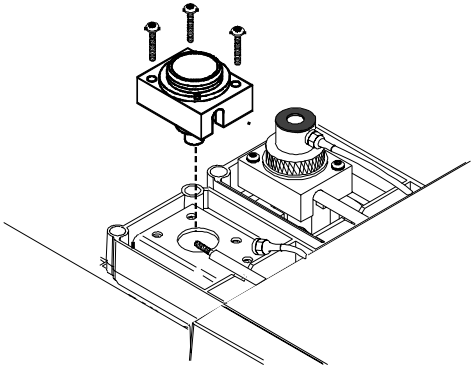
Procedure: Reassembling the detector

Caution Handle the clean collector and insulators only with forceps (or tweezers)!

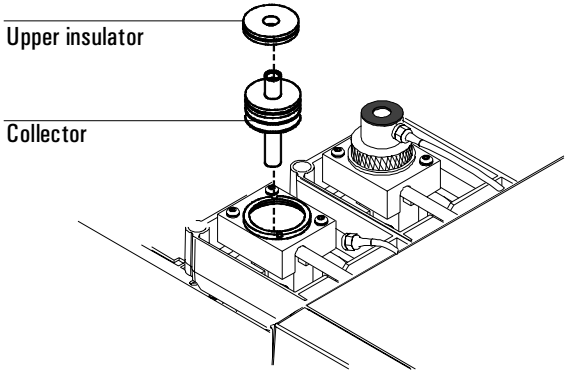
Materials needed:

- Forceps or tweezers
- T-20 Torx screwdriver

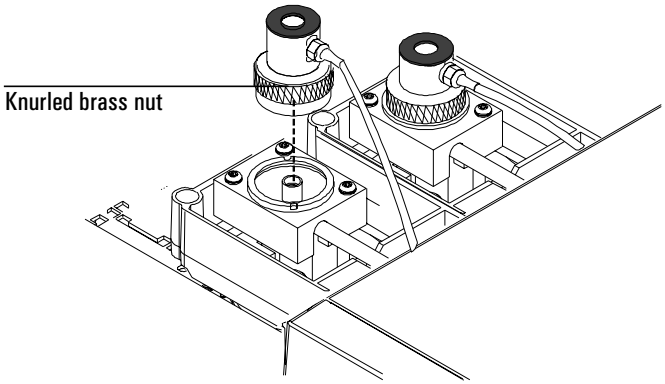
1. Insert the lower insulator into the lower collector assembly. Install the lower collector assembly and tighten the three screws.



2. Replace the collector and install the upper Teflon insulator.



3. Install the upper collector assembly and tighten the knurled nut finger-tight.



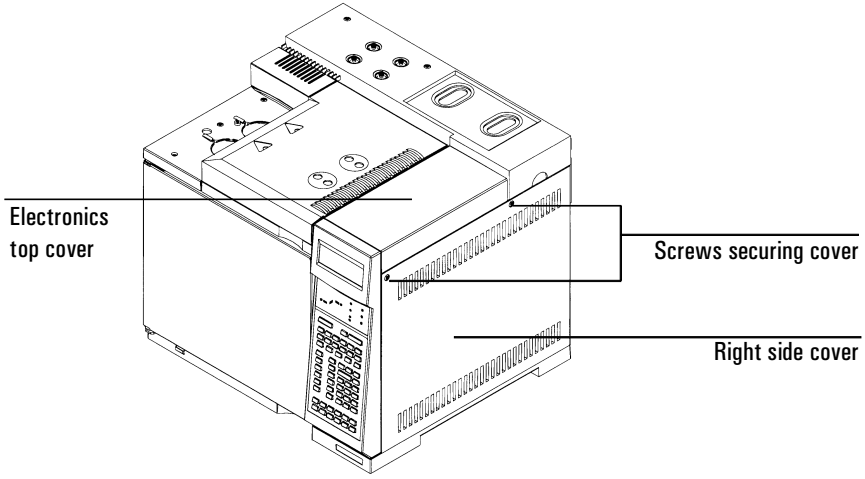
4. Close the GC detector cover. You can now restore normal operating conditions.

Procedure: Replacing the FID ignition wire

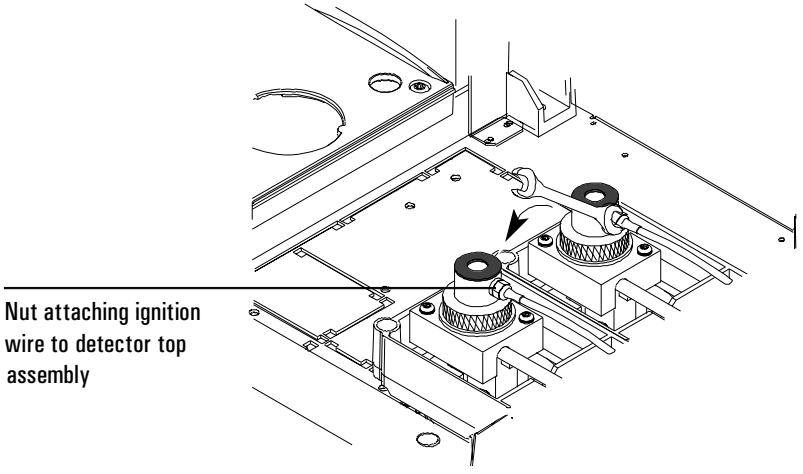
Materials needed:

- 5/16-inch wrench
- T-20 Torx screwdriver
- ESD wrist strap
- New ignition wire assembly (part no. G1531-60680)

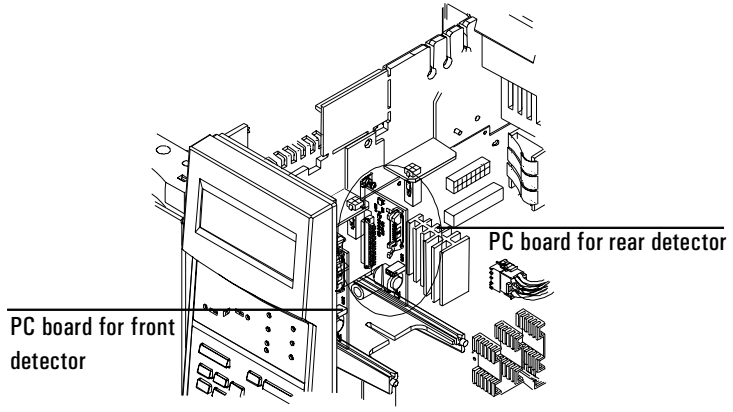
1. Complete the following preliminary steps:
   - Allow the detector to cool to room temperature. When the detector is cool, turn off the GC.
   - Lift the GC detector cover to access the FID.
   - Remove the electronics top cover.
2. Remove the two screws securing the right side cover and remove the cover.



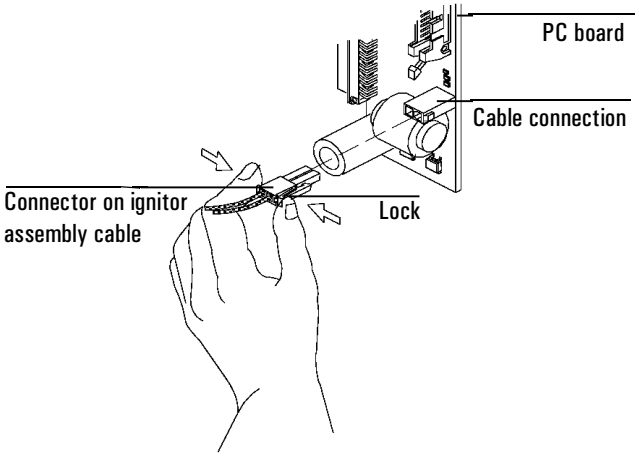
3. Using the wrench, loosen the ignition wire from the detector top assembly. Disconnect the wire completely. Do not lose the small copper washer between the top assembly and the ignition wire connection.



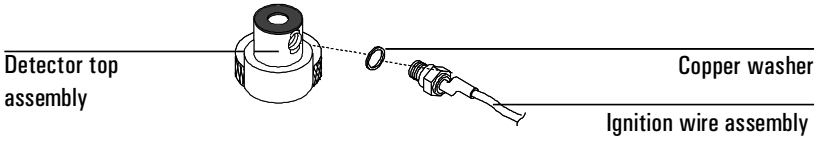
4. The other end of the ignition cable is connected to the detector PC board. Use the figure below to locate the PCB. Make sure to put on the ESD wrist strap at this time and connect it to a proper ground.



5. To disconnect the cable connection, squeeze the lock and gently pull the connector free. Attach the new ignitor cable by squeezing the lock and sliding the connector into the slot.



6. Place the copper washer on the other end of the ignition cable. Attach the other end of the ignition cable to the detector top assembly, and finger-tighten the screw to snugness. Then use the screwdriver to tighten the screw firmly.

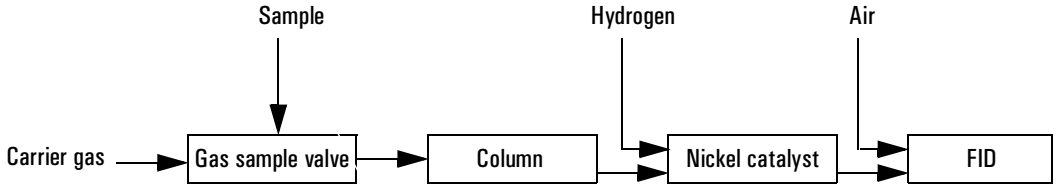


7. Replace the right side cover and the two screws. Replace the electronics top cover.

8. Turn on the GC and restore normal operating conditions.

The Nickel Catalyst Tube

The Nickel Catalyst Tube accessory, G2747A, is used for trace analysis of CO and CO_2 with a flame ionization detector. The gas sample is separated on the column and passed over the hot catalyst in the presence of hydrogen, which converts the CO and CO_2 peaks to CH_4.



Gas flows

For a standard FID installation:

Gas	Flow rate, mL/min
Carrier (helium)	30
FID hydrogen	30 (see Caution)
FID air	400

For a TCD/FID in-series installation:

Gas	Flow rate, mL/min
Carrier (helium)	30
TCD switching flow	25
FID hydrogen	45 (see Caution)
FID air	500

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

The nickel catalyst can be damaged by exposure to air.

Temperature

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

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

Repacking the catalyst

The nickel catalyst can be damaged by exposure to air or by impurities in the samples or gases. If performance is significantly degraded, repack the catalyst tube.

WARNING Hydrogen (H_2) is flammable and is an explosion hazard when mixed with air in an enclosed space (for example, the oven). In any application using H_2, turn off the supply at its source before working on the instrument.

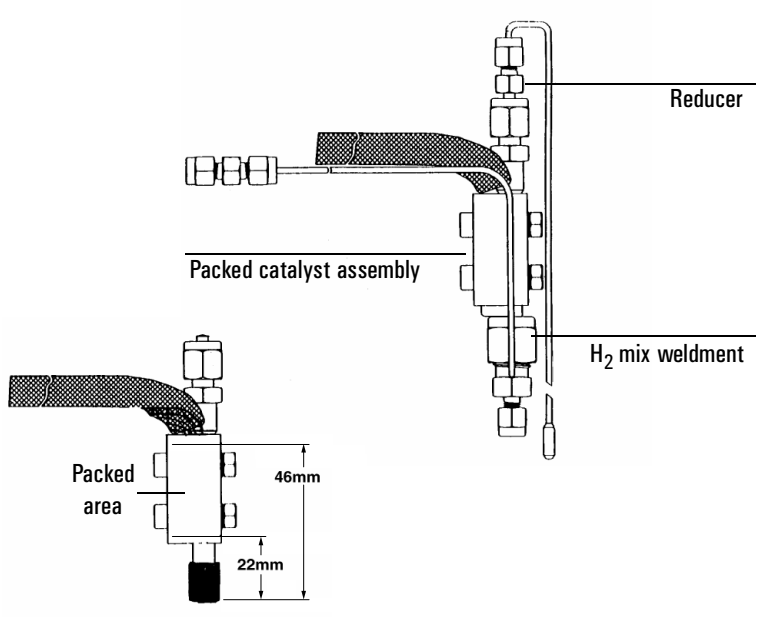
WARNING Both nickel oxide and some forms of silicon oxide are considered carcinogens for humans. Perform all work in a fume hood and wear cotton gloves at all times. Remove any spills with an HEPA-type vacuum cleaner, avoiding any action that raises dust. Alert your company's Safety group if a spill occurs.

WARNING Due to the possibility of dermatitis, wash the arms and hands with soap and water after use. Long sleeves are recommended during any use and spill cleanup. If long sleeves are not worn, long gloves are an acceptable substitute.

Caution Be sure to read the Material Safety Data Sheet (MSDS) provided with the catalyst before performing this procedure.

1. Turn off the back inlet thermal zone. Turn off all other heaters. When the catalyst tube has cooled to room temperature, turn off the power to the GC and disconnect the power cord. Bleed down the residual hydrogen and carrier gas pressures.
2. Remove the three screws holding the cover plate on top of the catalyst tube. Remove the plate and the insulation around the NCT.
3. From inside the oven, loosen the two screws holding the insulation cup. Remove the cup and insulation.

4. Use two wrenches to disconnect the H_2 mix weldment from the bottom of the catalyst assembly. Be careful NOT to place stress on the 1/16-inch tube. Stress can damage the weldment.



5. Use two wrenches to remove the reducer on the top of the catalyst assembly.
6. Gently lift the catalyst assembly out of the injection area. Both ends of the catalyst tube are now accessible.
7. Use a hooked instrument to remove the glass wool plug from the bottom of the tube. Make sure you get all of it.
8. Empty the old catalyst from the tube (you may have to break it out with a pointed tool). Make sure you get it all out.
9. Use a thin rod to push out the top glass wool plug from the tube.
10. Clean the inside of the tube thoroughly with methanol. Do not use any sharp metal tools on the inside of the tube. A cotton swab carefully used will ensure cleanliness. Dry the tube.

11. The previous figure shows the dimensions for repacking the tube correctly. If any catalyst is outside the heated zone, severe tailing of CO will result.

    Prepare a simple depth gauge using a wooden cotton swab or any other handy rod or tubing. Use tape or paint to mark the stick at 46 mm from the blunt end and at 22 mm from the blunt end.

12. Roll up a piece of glass wool about the size of a large pea. Push this into the tube from the 1/4-inch end and seat it firmly. Measure the depth of this glass wool with the depth gauge—it should be 46 mm from the end of the tube. If necessary, add more glass wool. A slight compression of the glass wool during the measurement works best.

13. Turn the catalyst assembly upside down and add catalyst slowly. Tap gently to help seat it. When the catalyst is 22 mm from the end, stop adding catalyst. Do NOT crush the catalyst when packing or measuring the depth.

14. Add a single glass wool plug to fill the remaining part of the tube to within 5 mm of the end. This plug should be gently compressed during installation.

Caution Before installing the catalyst assembly into the oven, carefully wipe it to remove any catalyst dust.

15. Reassembly is the reverse of steps 1 through 6. Make sure that the insulation is carefully repacked around the tube before you reinstall the injector cover plate and the insulation cup.

16. Leak test the new installation.

WARNING Hydrogen (H_2) is flammable and is an explosion hazard when mixed with air and confined in an enclosed space (for example, the oven).

17. Start the carrier and hydrogen flows. Allow them to flow for 15 minutes.

18. Heat the nickel catalyst to 375°C and hold for 30 minutes. The accessory is ready for use.

23 The Thermal Conductivity Detector

General Information

TCD pneumatics

Conditions that prevent the detector from operating

Filament passivation

Carrier, reference, and makeup gas

Negative polarity

Analyzing for hydrogen

Operating the TCD

Columns and Traps

Gas pressures

Operating the TCD

Procedure: Using the TCD

Checkout Conditions and Chromatogram

TCD checkout conditions

Typical TCD checkout chromatogram

Maintaining a Thermal Conductivity Detector

Correcting TCD performance problems

Procedure: Thermal cleaning

The Thermal Conductivity Detector

General Information

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

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

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

Because the TCD does not destroy the sample during the detection process, this detector can be hooked up in series to a flame ionization detector or other detector.

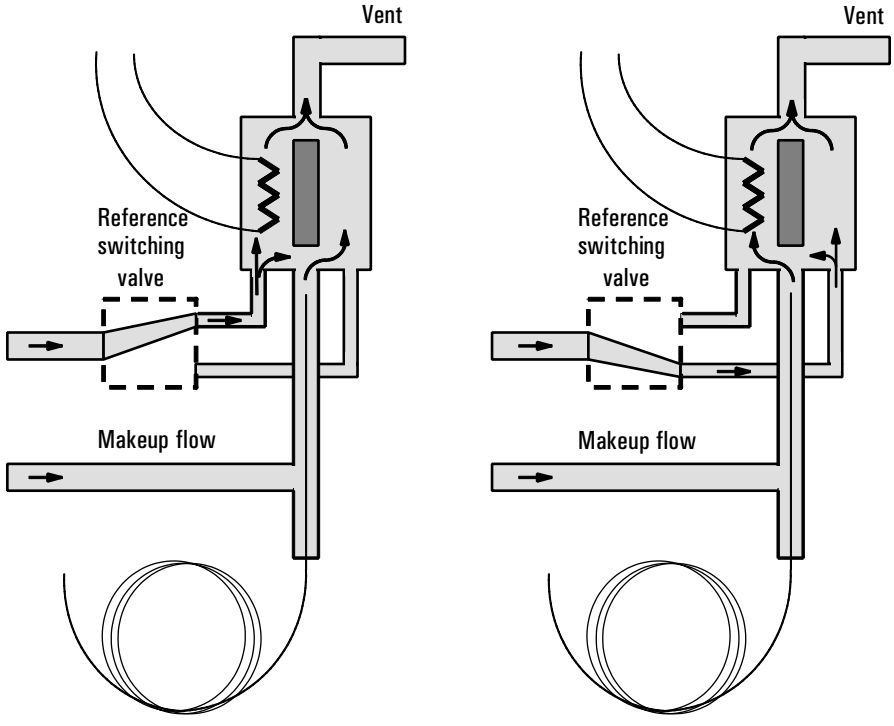


Figure 76. TCD — Conceptual diagram

TCD pneumatics

Figure 77 shows the pneumatics design of the TCD.

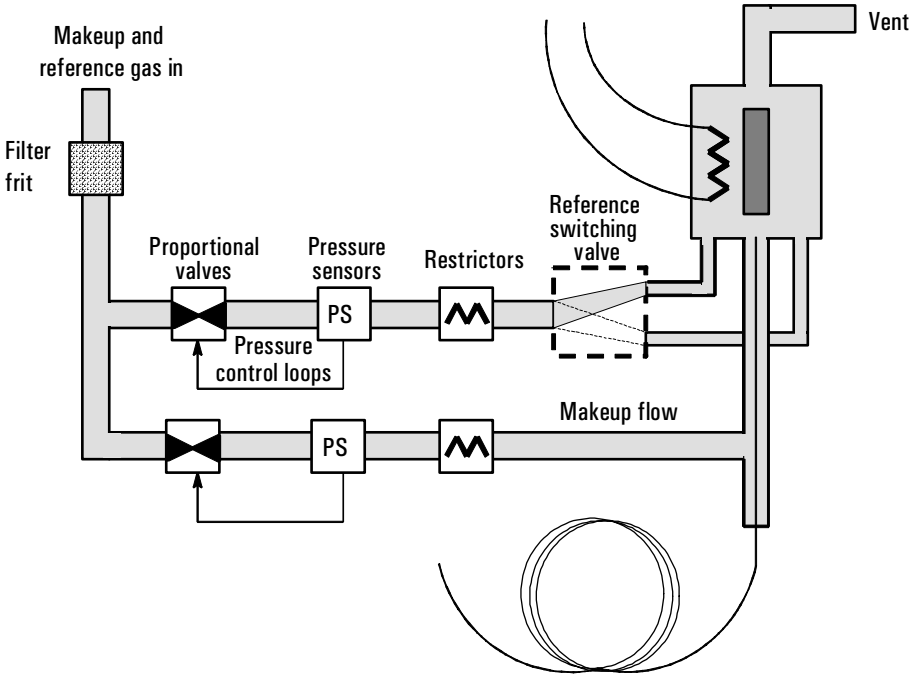


Figure 77. TCD pneumatics

Conditions that prevent the detector from operating

- Temperature set below 150°C
- Broken or shorted filament
- Reference gas flow set to 0

Filament passivation

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

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

Carrier, reference, and makeup gas

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

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

Use Figure 78 to select a value for reference gas flow for either capillary or packed columns. Any ratio within ±0.25 of that in the figure is suitable.

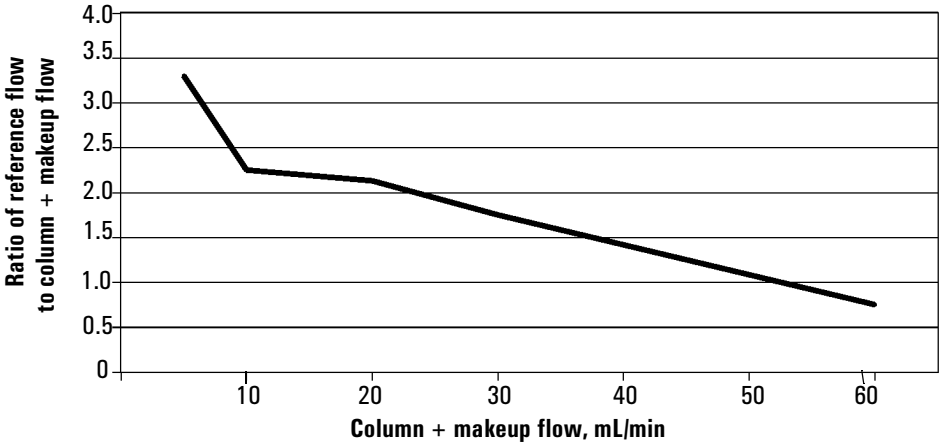


Figure 78. Selecting the reference gas flow

Negative polarity

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

`Neg polarity ON` inverts the peak so the integrator or ChemStation can measure it. `Neg polarity` can be a run table entry; see "Run time programming".

Analyzing for hydrogen

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

There are two solutions to this problem:

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

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

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

Operating the TCD

Use the information in Table 62 when selecting temperatures and flows for the TCD. Use Figure 79 to locate minimum source pressures. If you have an EPC detector, you must add 10 psi (69kPa) to the source pressure on t he chart.

Table 62. Recommended Flow Rates and Temperatures

Gas type	Flow range
Carrier gas *(hydrogen, helium, nitrogen)*	Packed, 10 to 60 mL/min Capillary, 1 to 5 mL/min
Reference *(same gas type as carrier)*	15 to 60 mL/min See Figure 79 to select a value.
Capillary makeup *(same gas type as carrier)*	5 to 15 mL/min—capillary columns 2 to 3 mL/min—packed columns
Detector temperature	
< 150° C, cannot turn on filament Detector temperature should be 30°C to 50°C greater than highest oven ramp temperature.	

Gas pressures

Choose a flow, find a pressure, set source pressure 10 psi (70 kPa) higher.

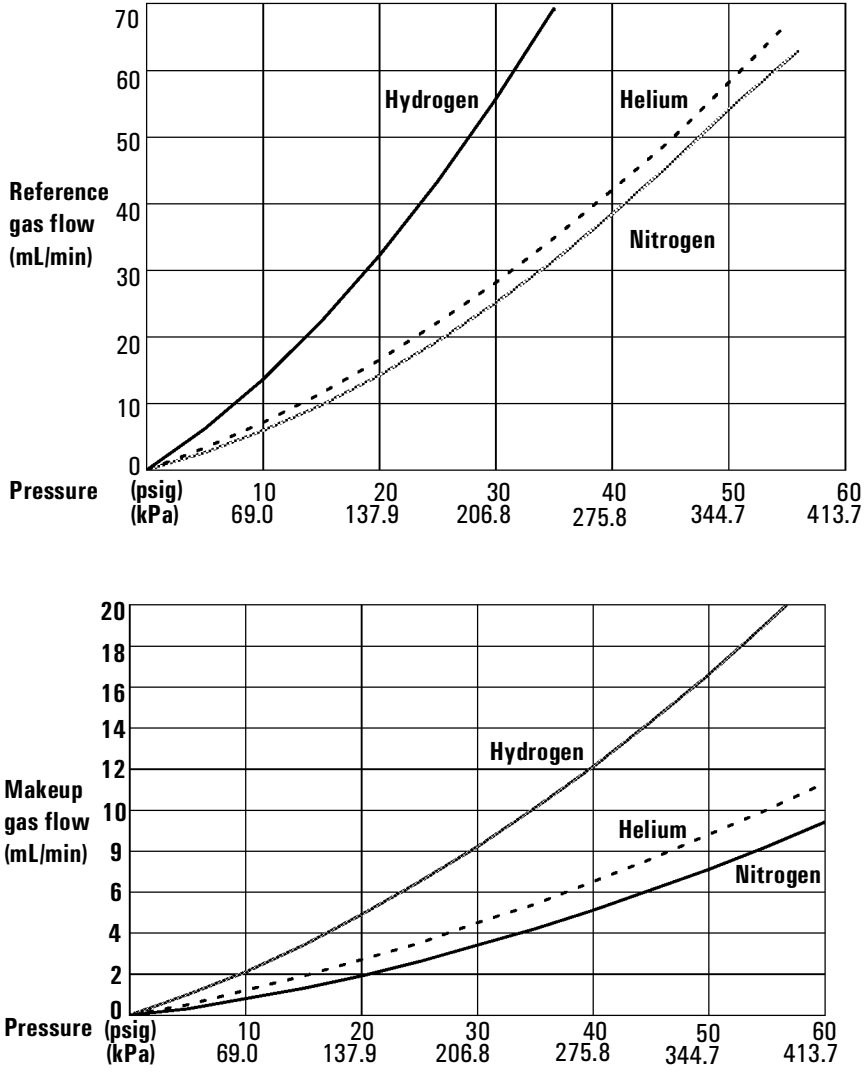
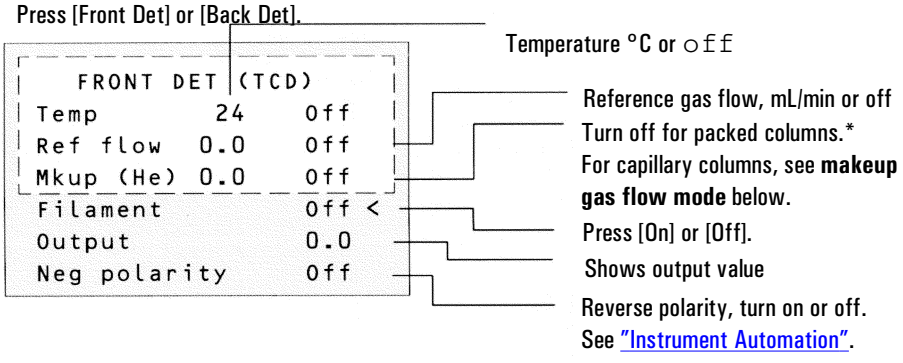


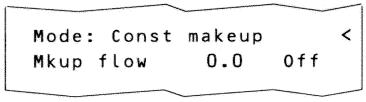
Figure 79. Typical pressure/flow relationships, reference and makeup gases *(at 25°C and 1 atmosphere of pressure)*

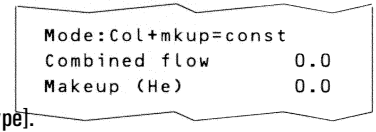
Operating the TCD



Makeup gas flow mode:

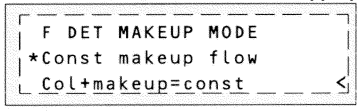
If configured for capillary columns, your control table will also include one of these:



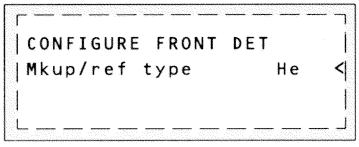


To **change makeup mode**, scroll to Mode: and press [Mode/Type].

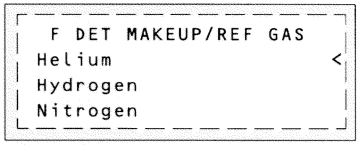
Make a selection and enter the appropriate flow values.



To **view makeup/reference gas,** press [Config][Front Det] or [Config][Back Det]:



To **change makeup/reference gas,** press [Mode/Type]:



Select the appropriate gas.

* A makeup flow of 2 to 3 mL/min improves peak shapes.

Figure 80. TCD control table

Procedure: Using the TCD

This procedure assumes that detector support gases are connected, the system is leak-free, and a column is installed. Before operating the detector, set oven temperature, inlet temperature, and inlet/column flow.

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

Caution Detector electronics depend on the correct gas type configuration.

4. Set the reference gas flow rate.
5. If you are using ***packed columns***, turn off the makeup gas (or proceed to Step 6 and enter 2 to 3 mL/min, see "Carrier, reference, and makeup gas") and proceed to Step 7.
6. If you are using ***capillary columns:***
   a. If your column is *defined* and connected to an EPC inlet, choose a new flow mode ("Makeup gas flow") if desired, and set the makeup gas flow or combined flow.
   b. If your column is connected to a nonEPC inlet, enter a makeup gas flow. Only constant flow is available in this case.
7. Turn on the filament. Allow about 30 minutes for thermal stabilization. A longer period may be needed for the highest sensitivity.
8. If necessary, turn `Negative polarity` [On] to invert negative-going peaks. When a sample contains components giving both positive- and negative-going peaks, `Neg polarity` can be switched on and off during a run as a timetable event.

Short-cut procedure:
(assumes your setpoints are stored)

1. Open detector control table.
2. Turn temperature `On`.
3. Turn makeup gas `On`, if needed.
4. Press [Det Control]
5. Press [On]

Checkout Conditions and Chromatogram

This section contains a typical examples of a test sample chromatogram. It may be used as a general guide to instrument performance.

Note that injection volumes listed with operating conditions do not necessarily indicate total absolute volume injected. Volume given is simply the graduation (plunger position) read from a standard 10 µL syringe. For a heated inlet, actual sample volume injected will also include an additional 0.4-0.7 µL, the volume of sample volatilized from inside the syringe needle. For the dedicated, on-column inlet (unheated), the syringe plunger position more accurately reflects the true injected volume.

Also note that the following procedure and results are intended only to provide evidence of a properly functioning inlet and/or detector system; they are not necessarily suitable to test a given system against its specification limits.

TCD checkout conditions

Column and sample

Type	HP-5 30m × 0.32mm × 0.25 µm PN 19091J-413
Sample	FID Checkout 18710-60170
Injection volume	1 µL

Inlet

Temperature	250° C Purged/Packed or Split/Splitless
	Oven Track Cool On-Column
	40°C PTV (see below)
Inlet pressure	25 psi (Constant pressure for EPC inlets, helium)

Split/Splitless

Mode	Splitless
Purge flow	60 mL/min
Purge time	0.75 min

Inlet, continued

PTV

Mode	Splitless
Inlet temperature	40°C
Initial time	0.1 min
Rate 1	720°C/min
Final temp 1	350°C
Final time 1	2 min
Rate 2	100°C/min
Final temp 2	250°C
Final time 2	0 min
Inlet pressure	25 psi (constant pressure for EPV inlets)
Purge time	0.75 min
Purge flow	60 mL/min

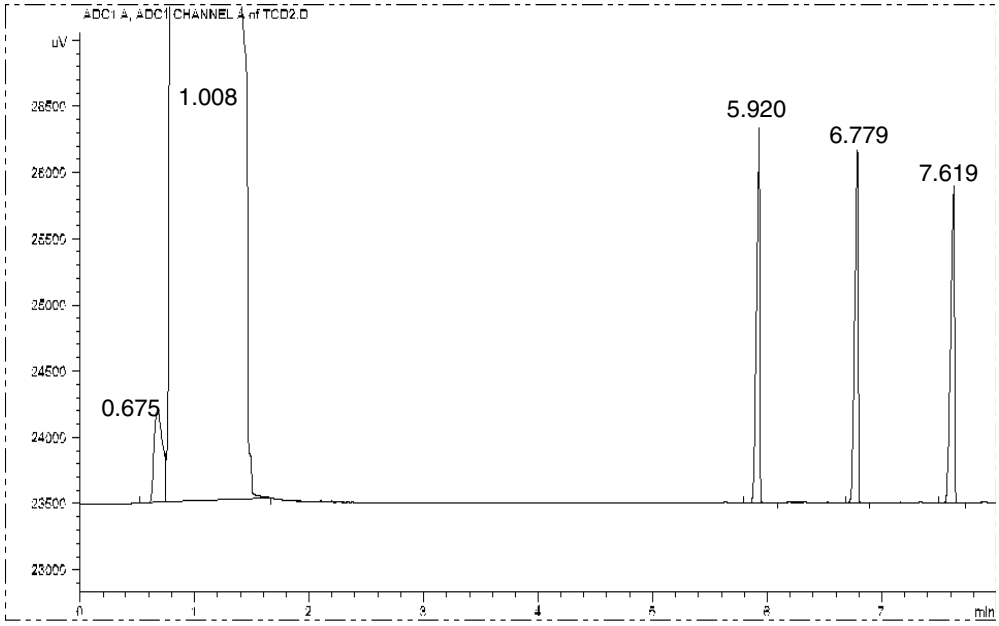
Detector

Temperature	300° C
Reference flow (He)	30 mL/min
Makeup flow (He)	2 mL/min
Offset	Should be < 30 display counts

Oven

Initial temp	40° C
Initial time	0 min
Rate 1	25° C/min
Final temp	90° C
Final time	0 min
Rate 2	15° C/min
Final temp	170° C
Final time	2 min

Typical TCD checkout chromatogram



Your retention times will differ, but peaks should resemble this example.

Maintaining a Thermal Conductivity Detector

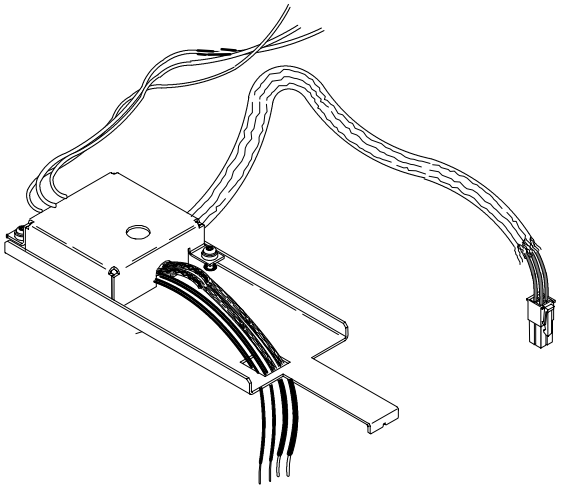


Figure 81. The TCD

Correcting TCD performance problems

If the TCD is displaying problems such as a wandering baseline, increased noise level, or changes in response on a checkout chromatogram, it is probably contaminated with deposits from such things as column bleed or dirty samples.

The TCD is cleaned by a process known as bakeout. Bakeout should be performed only after you have confirmed that the carrier gas and the flow system components are leak and contaminant free.

Caution Baking out the detector with a large air leak present can destroy the filament.

Procedure: Thermal cleaning

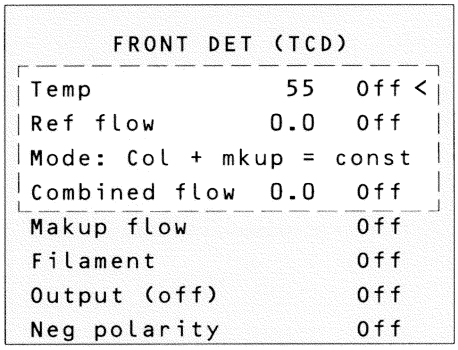
The only common maintenance procedure you will need to perform on the TCD is thermal cleaning.

The TCD can become contaminated with deposits from such things as column bleed or dirty samples. A wandering baseline, increased noise level, or changes in response on a checkout chromatogram all indicate contamination.

Thermal cleaning is also known as **bakeout**. Bakeout should be performed only after you have confirmed that the carrier gas and the flow system components are leak and contaminant free.

Caution You must turn off the TCD and cap the detector column fitting to prevent irreparable damage to the filament caused by oxygen entering the detector.

1. Turn the detector off.
2. Remove the column from the detector and cap the detector column fitting.
3. Set the reference gas flow rate between 20 and 30 mL/min. Set the detector temperature to 400°C.



4. Allow thermal cleaning to continue for several hours. Then cool the system to normal operating temperatures.

24 The Nitrogen-Phosphorus Detector

General Information

Software requirements

NPD pneumatics

Conditions that prevent the NPD from operating

Gas purity

The bead

- Adjust offset
- Aborting adjust offset
- Turning off the detector
- Setting adjust offset on the clock table
- Equilibration time
- Procedure: Changing equilibration time
- Turning hydrogen off during a solvent peak
- Turning hydrogen off between runs
- Bead voltage
- Extending the life of the bead

Temperature programming

Electrometer

- Data rates
- Procedure: Setting data rate for NPD

Jets and collectors

Operating the NPD

Columns and Traps

Gas pressures

Operating with EPC

- Procedure: Using the NPD

Checkout Conditions and Chromatogram

NPD checkout conditions

NPD checkout chromatogram

Maintaining a Nitrogen-Phosphorus Detector

NPD illustrated parts breakdown

Correcting NPD hardware problems

- Procedure: Replacing the bead assembly
- Procedure: Cleaning detector and collector; changing insulators and rings

Replacing or cleaning the jet

- Procedure: Removing and inspecting the jet
- Procedure: Cleaning the jet
- Procedure: Replacing the jet and reassembling the detector

The Nitrogen-Phosphorus Detector

General Information

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

Software requirements

This discussion assumes that the following firmware/software is installed:

Product	Software/firmware revision
6890 GC	A.03.03 or higher
Agilent GC ChemStation	A.05.02 or higher
Agilent MSD ChemStation	G1701AA or higher

Software/firmware with numbers less than shown in the table will cause reduced bead lifetime. See Agilent service for updates.

NPD pneumatics

Figure 82 shows the flow paths for the NPD.

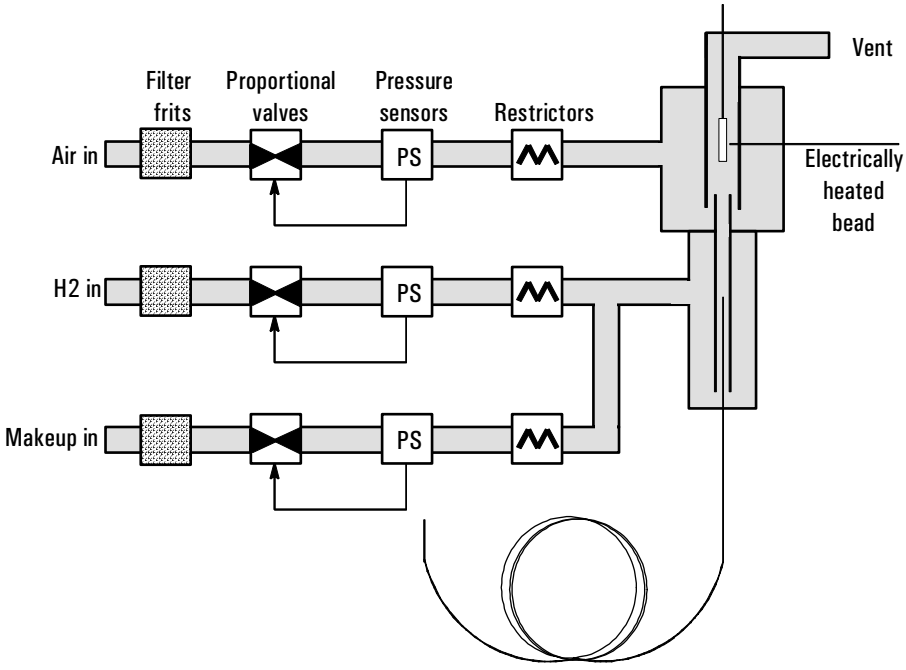


Figure 82. NPD pneumatics

Conditions that prevent the NPD from operating

- Hydrogen or air setpoints are set to 0.
- If the detector temperature is below 150°C or the oven is off, the `Adjust offset` process will not start.

Gas purity

Because of its high sensitivity, the NPD requires very pure gases. We strongly recommend that moisture and organics traps be used on the carrier gas and all detector gases, including the detector hydrogen, air, and makeup gases.

The bead

Two ceramic beads are available:

Bead color	Part no.	Advantages	Disadvantages
White	G1530-60570	Standard	Phosphorus tails
Black	5183-2007	Durable, no phosphorus tailing	Lower nitrogen sensitivity, about 40%

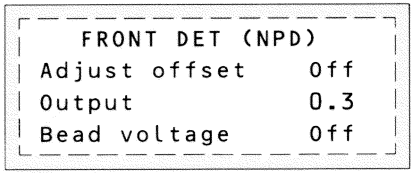
There are three setpoints associated with the bead—`Adjust offset`, `Bead voltage`, and `Equib time`.

Adjust offset

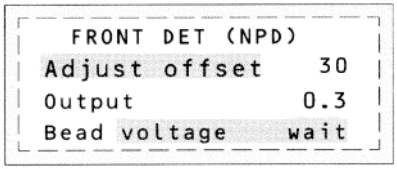
When you enter a value here, or press [On] to use the stored value, detector gas flows turn on, the bead heats, and the bead voltage adjusts until `Output` is stable and equal to the entered value. There are five stages of `Adjust offset`.

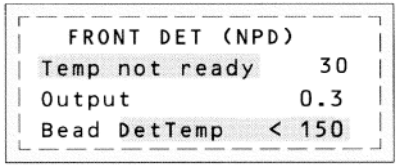
Detector off. When the detector is off, `Adjust offset` and `Bead voltage` are `Off` and initial `Output` is displayed.

Press [Front Det] [Det Control] or [Back Det] [Det control].

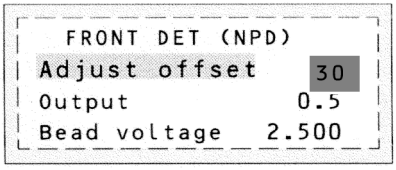


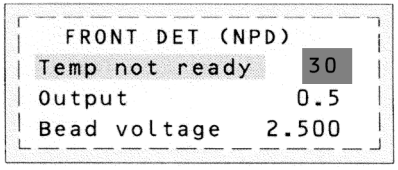
Detector on—detector temperature less than 150°C. When you enter an `Adjust offset` value or press [On], detector gases are off and the display blinks the following messages:



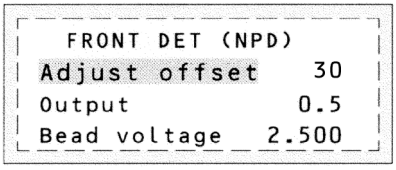


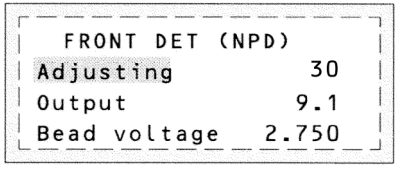
Detector on—waiting for oven and/or detector to reach temperature setpoint and equilibrium. When the detector temperature exceeds 150°C, the hydrogen and air flows turn on and the bead begins to heat while the oven and detector reach setpoint and equilibrate. The display blinks:



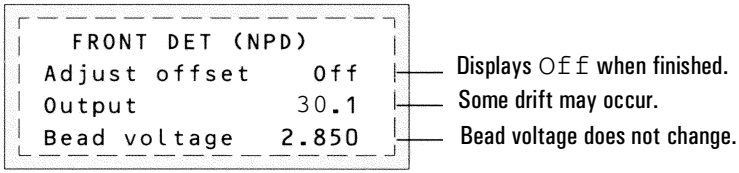


Detector on—during adjust offset and equilibration time. When the detector and oven temperatures reach setpoint and equilibrate, the `Adjust offset` process begins. The bead voltage is increased until the output is close to the `Adjust offset` value. Equilibration time (see "Equilibration time") begins. The display blinks.





Detector on and ready. When the Adjust offset value is reached and the equilibration time has passed, the Adjust offset line is Off. Your detector is on and ready.



Aborting adjust offset

Press [Delete] with the cursor on the Adjust offset line. This cancels the adjustment without turning off the detector gases and bead voltage. This is useful if you wish to start a run before the bead equilibration time is passed.

Turning off the detector

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

Setting adjust offset on the clock table

You can use the Clock table feature to turn the Adjust offset on at a specified time. Details can be found in "Clock time programming".

Caution It is not recommended that you Adjust offset between runs. Before the oven reaches its initial setpoint and the system is thermally stable, column bleed and residual peak tailing can mask an otherwise stable baseline. This can waste time between runs.

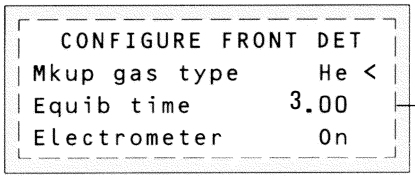
Equilibration time

Equilibration time begins when Output nears the Adjust offset value. During equilibration, Output is measured and compared to the Adjust offset value. If Output stays close to the Adjust offset for the entire equilibration time, the detector becomes ready. However, if the Output is too high or too low at any time during the equilibration period, the adjust offset process continues and the equilibration time begins again.

We recommend an equilibration time of 0.0 minutes and the automatic `Adjust offset` process. Some beads do not respond well to the automatic process. For these, we suggest starting at 2.0 volts and bringing up the bead voltage gradually, 10 mV at a time, until the desired offset is reached.

Procedure: Changing equilibration time

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



2. Enter a value (in minutes). Long equilibration time reduce bead lifetime.

Turning hydrogen off during a solvent peak

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

Turning hydrogen off between runs

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

Bead voltage

`Bead voltage` shows the voltage used to heat the bead. It can be an actual value, dependent on the `Adjust offset` value, or can be entered as a setpoint.

Equilibration time is not used when you enter a setpoint for `Bead voltage`, so you cannot estimate your baseline stability. Use the `Bead voltage` setpoint when the automatic startup does not work.

`Bead voltage` is also useful for small adjustments between runs. If you observe a baseline drift, you can enter a small, one-time change to compensate for the drift without having to wait for the `Equib time`.

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

Extending the life of the bead

- Use the lowest practical adjust offset or bead voltage.
- Run clean samples.
- Turn the bead off when not in use.
- Keep the detector temperature high (320 to 335°C).
- Turn the hydrogen flow off during solvent peaks and between runs.
- If your NPD is `Off` for a long time in a high-humidity environment, water may accumulate in your detector. To evaporate this water:
  a. Set the detector temperature at 100°C and maintain it for 30 minutes.
  b. Set the detector temperature to 150°C and maintain it for another 30 minutes.

Temperature programming

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

- Set the carrier gas in the `Constant flow` mode. Set detector makeup gas to `Const makeup`.
- If you choose to work in the constant pressure mode, the makeup gas should be set in the `Col+mkup=const mode.`

Electrometer

The Configure Detector control table contains an `On/Off` setpoint for the `Electrometer`. You do not need to turn the electrometer on and off when operating your NPD.

Caution Do not turn off the electrometer during a run. It will turn off the detector `Output`.

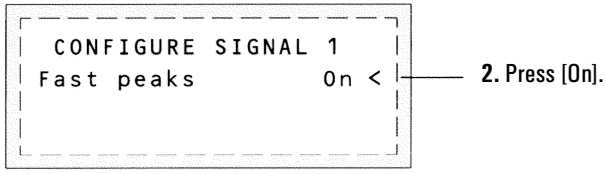
Data rates

Analog output for the NPD can be presented at either of two speeds. The faster speed allows minimum peak widths of 0.004 minutes, while the standard speed allows peak widths of 0.01 minutes.

Procedure: Setting data rate for NPD

If you are using the *fast peaks* feature, your integrator must be fast enough to process data coming from the GC. Integrator bandwidth should be at least 15 Hz. To use fast peaks:

1. Press [Config][Signal 1] or [Config][Signal 2].



Digital output to the ChemStation is available at eleven speeds ranging from 0.1 Hz to 200 Hz, capable of handling peaks from 0.001 to 2 minutes wide. Consult "Signal Handling".

The *fast peaks* feature does not apply to digital output.

Jets and collectors

The *capillary optimized* NPD is only used with capillary columns. It is shipped with the standard jet and collector.

Table 63. Jets and Collectors for the Capillary-Optimized NPD

Type	Part no.	id	Use with
Standard jet	G1531-80560	0.29 mm	Either collector
Extended jet (optional)	G1534-80580		Either collector
Standard collector	G1534-20530	7 mm	
Small id collector (optional)	G1534-20660	5 mm	

The *adaptable* NPD fits packed columns and can be adapted to fit capillary columns. It is shipped with the capillary column jet and standard collector. You must change the jet to use packed columns. Instructions appear on "Replacing or cleaning the jet".

Table 64. Jets and Collectors for the Adaptable NPD

Type	Part no.	id	Use with
Capillary column jet	19244-80560	0.29 mm	Either collector
Extended jet	G1534-80590		Either collector
Standard collector	G1534-20530	7 mm	
Small id collector	G1534-20660	5 mm	

The extended jets, used with the small id collectors, reduce the exposure of the sample to heated metal and reduce tailing of some very polar components.

Operating the NPD

Use the information in Table 65 to select temperatures and flows. Choose a minimum source pressure from Figure 83. You must add 10 psi (69 kPa) to the source pressure on the chart.

Table 65. Flows, Temperatures, and Bead Information

Gas type	Recommended flow
Carrier gas *(helium, hydrogen*, nitrogen)*	*Capillary*, choose optimum flow based on column dimensions.
Detector gases	
Hydrogen	3.0 mL/min (maximum flow is 5 mL/min)
Air	60 mL/min
Capillary makeup *(helium, ** nitrogen)*	Nitrogen: 5 to 10 mL/min Helium: less than 5 mL/min
Temperature (Default is 250° C; range is ambient to 400° C) < 150° × C, the `Adjust offset` process will not start. 325 to 335°C is recommended. Detector temperature should be greater than the highest oven ramp temperature. With higher detector temperatures, less bead heating voltage is required.	
Adjust offset Default is 30 pA, suggested operating range is 30 to 40 pA, and allowable range is 10 to 99 pA. ≥50 pA increases sensitivity but reduces bead life. Lower settings reduce sensitivity and increase bead life, but too low will result in solvent quenching. Once Adjust offset is turned on, allow 20 to 60 minutes for detector to reach readiness.	
Equib time (Default is 5 minutes; range is 0 to 999.9 minutes) Recommended time is 0.0 minutes.	
Bead voltage (range is 0 to 4.095 V) Use to make minor adjustments or manually activate the bead. Set Equib time = 0.0 and Bead voltage at 2.0. Increase voltage in 0.01 volt increments until the bead ignites.	

Gas pressures

Choose a flow, find a pressure, and set source pressure 10 psi (70 kPa) higher.

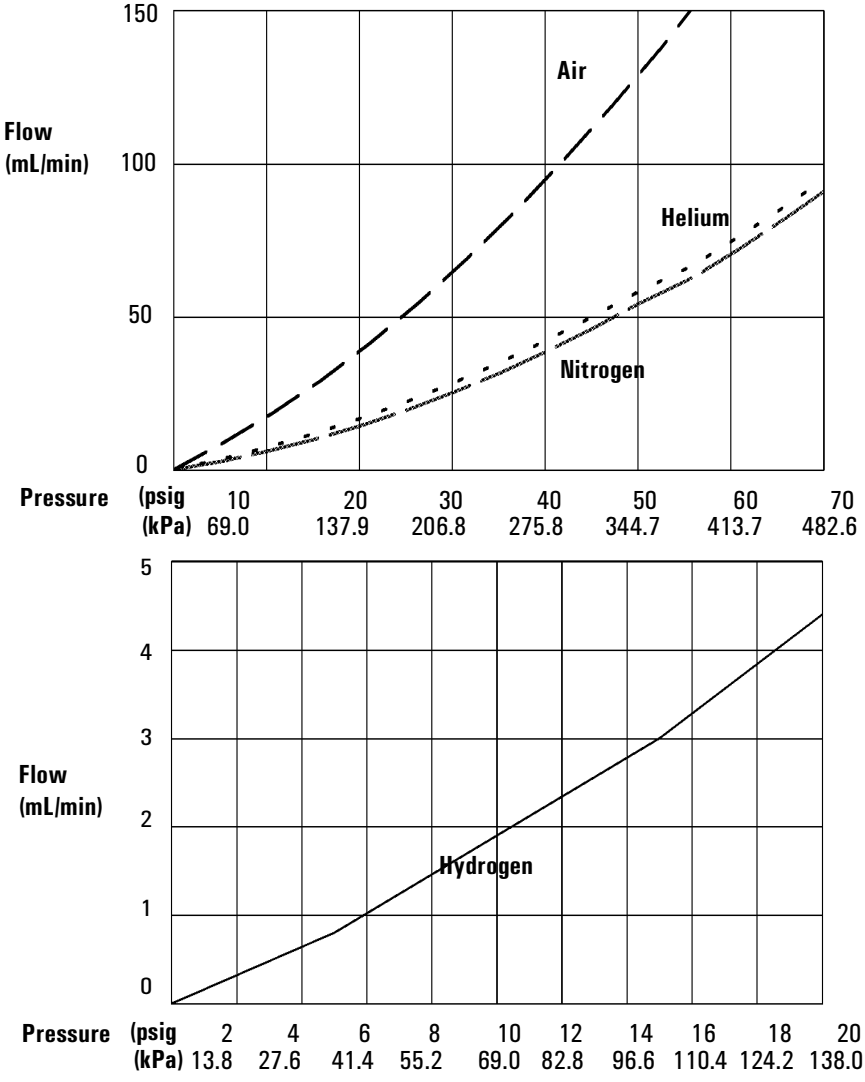
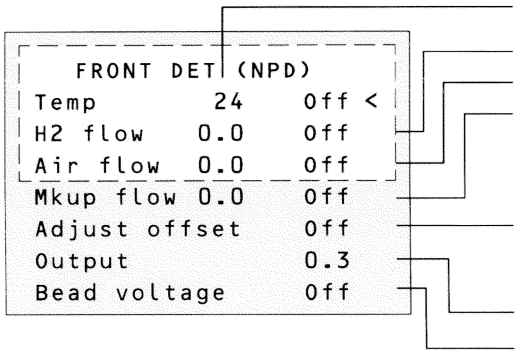


Figure 83. Pressure/flow relationships for NPD gases
(at 25°C and 1 atmosphere of pressure)

Operating with EPC

Press [Front Det] or [Back Det].



Temperature °C

Hydrogen flow, mL/min

Air flow, mL/min

Turn off for packed columns.
For capillary columns, see **makeup gas flow mode** below.

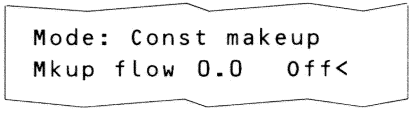
Adjust bead voltage automatically to achieve stable Output.
(10 to 99 pA).

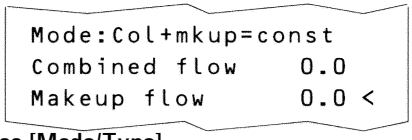
Actual value of detector output, pA

Actual bead heating voltage
(0 to 4.095 V)

Makeup gas flow mode

If column dimensions are specified, the control table will also include one of these:

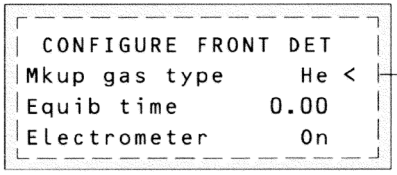




To **change makeup mode,** scroll to Mode: and press [Mode/Type].

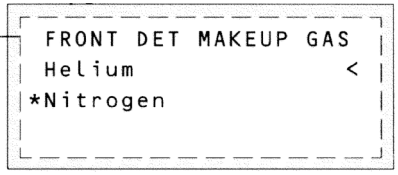
Make a selection and enter the appropriate flow values.

To change **makeup gas type** or **equilibration time,** press [Config][Front Det] or [Config][Back Det]:



You do not need to turn the electrometer on or off.

Press [Mode/Type] to **change makeup gas:**



Select the appropriate gas.

Figure 84. NPD control table

Procedure: Using the NPD

Before operating the NPD, make sure that detector gases are connected, a column is installed, the correct jet is installed, and the system is free of leaks. Set the oven temperature, inlet temperature, and column flow. Use the information in Figure 84 when editing the control tables.

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

1. Press [Config][Front Det] or [Config][Back Det].
   a. If you are using makeup gas, verify that the configured makeup gas type is the same as that plumbed to your instrument. Change the gas type, if necessary (page 511). Nitrogen is recommended.
   b. Check the equilibration time. The recommended value is 0.0.
2. Press [Front Det] or [Back Det] to open the NPD control table.
3. Set the detector temperature. The recommended range is 325 to 335°C.
4. Enter a hydrogen flow (3.0 is recommended), if desired, and press [Off].
5. Enter an air flow (60 is recommended), if desired, and press [Off].

   If you are using ***packed columns***, turn off makeup gas and proceed to step 7.

   If your ***capillary column*** is *defined* and connected to an EPC inlet, choose a new flow mode (page 511), if desired, and set the makeup gas flow. If you have set up your column in the constant flow mode, choose `const makeup`. If you have set up your column in the constant pressure mode, choose `Col+makeup=const`.

   If your column is *not defined*, or is connected to a non EPC inlet, enter a makeup gas flow. Only constant flow is available.
6. Enter `Adjust offset` number, or press [On] to begin the adjustment process. Your hydrogen and air flows will be switched on once the detector reaches 150°C.

Short-cut procedure:
(assumes correct setpoints are stored)

1. Open detector control table.
2. Turn temperature On.
3. Turn makeup gas On, if needed.
4. Press [Det Control].
5. Press [On].

Checkout Conditions and Chromatogram

This section contains a typical example of a test sample chromatogram. It may be used as a general guide to instrument performance.

Note that injection volumes listed with operating conditions do not necessarily indicate total absolute volume injected. Volume given is simply the graduation (plunger position) read from a standard 10 µL syringe. For a heated inlet, actual sample volume injected will also include an additional 0.4-0.7 µL, the volume of sample volatilized from inside the syringe needle. For the dedicated, on-column inlet (unheated), the syringe plunger position more accurately reflects the true injected volume.

Also note that the following procedure and results are intended only to provide evidence of a properly functioning inlet and/or detector system; they are not necessarily suitable to test a given system against its specification limits.

NPD checkout conditions

Column and sample

Type	HP-5 30m × 0.32mm × 0.25 μm PN 19091J-413
Sample	NPD Checkout 18789-60060
Injection volume	1 μL

Inlet

Temperature	200° C Purged/Packed or Split/Splitless
	Oven Track Cool On-Column
	60°C PTV (see below)
Inlet pressure	25 psi (Constant pressure for EPC inlets, helium)
Split/Splitless	
Mode	Splitless
Purge flow	60 mL/min
Purge time	0.75 min

Inlet, continued

PTV

Mode	Splitless
Inlet temperature	60°C
Initial time	0.1 min
Rate 1	720°C/min
Final temp 1	350°C
Final time 1	2 min
Rate 2	100°C/min
Final temp 2	250°C
Final time 2	0 min
Inlet pressure	25 psi (Constant pressure for EPC inlets)
Purge time	0.75 min
Purge flow	60 mL/min

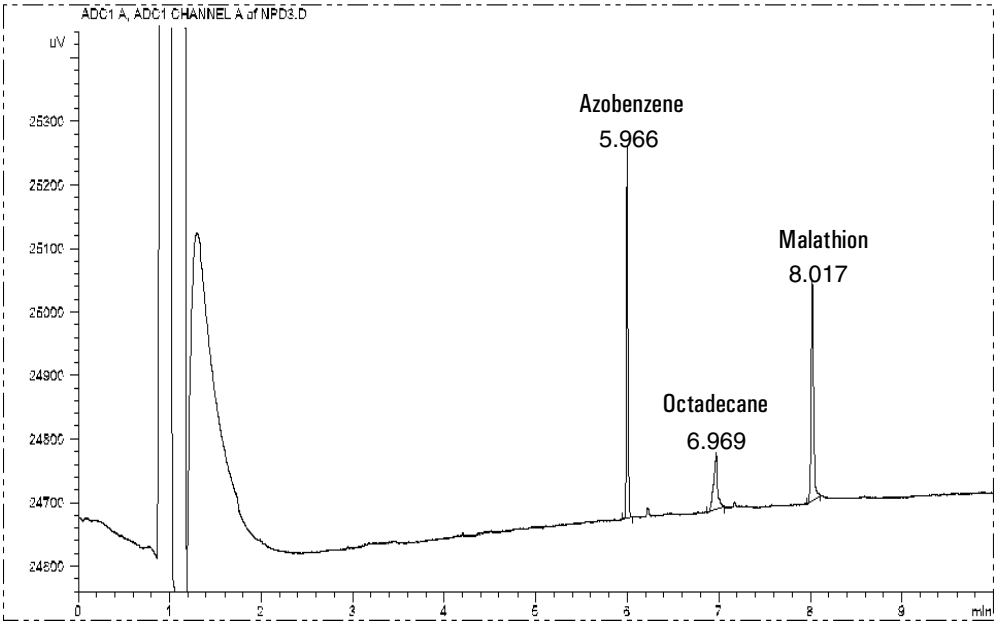
Detector

Temperature	300° C (325 to 330°C recommended)
H_2 flow	3 mL/min
Air flow	60 mL/min
Makeup + column flow	10 mL/min (nitrogen recommended)
Offset	50 pA (30 to 35 recommended)

Oven

Initial temp	60° C
Initial time	0 min
Rate 1	20° C/min
Final temp	200° C
Final time	3 min

Typical NPD checkout chromatogram



Your retention times will differ but peaks should resemble the example.

Maintaining a Nitrogen-Phosphorus Detector

NPD illustrated parts breakdown

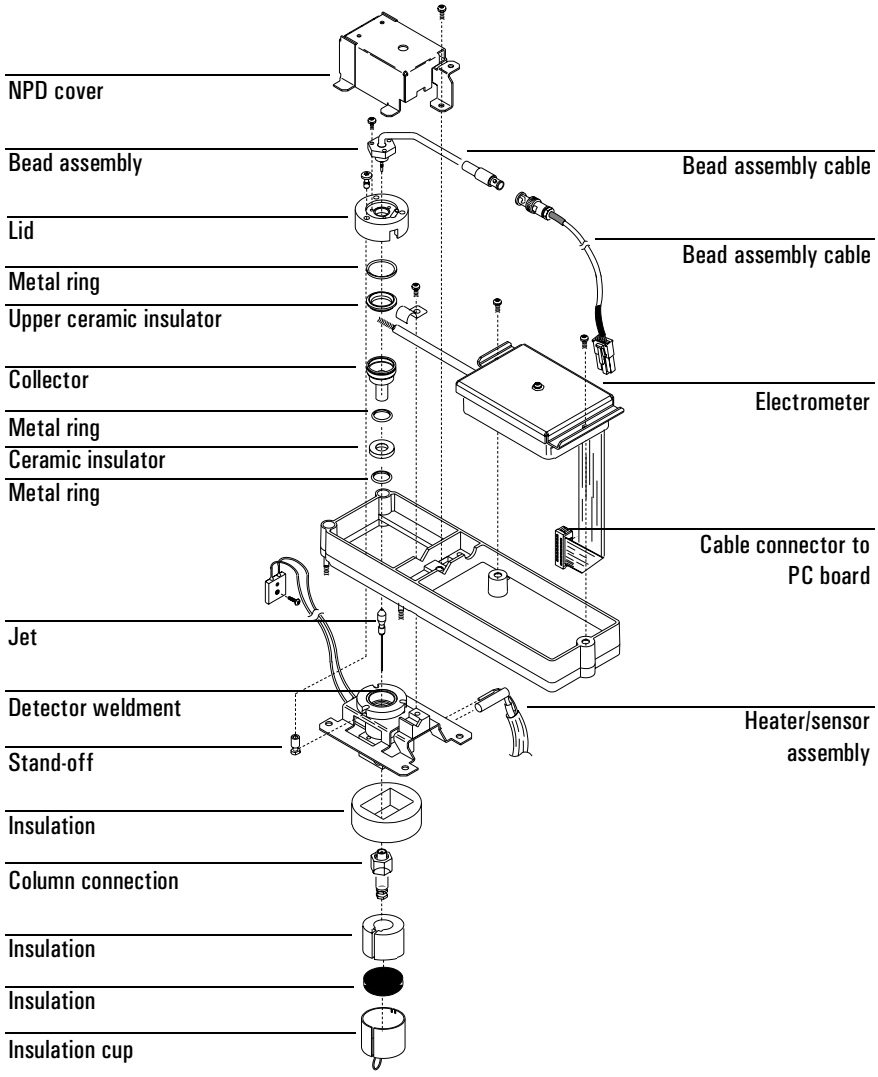


Figure 85. The NPD

Correcting NPD hardware problems

No detector response to injected sample

- A large concentration of solvent has extinguished the hydrogen/air plasma. Increase the bead voltage. Run the detector at a higher offset (for example, 40 to 50 pA), or use makeup gas at a flow rate of 5 mL/min.
- Check that hydrogen is flowing to the detector. Verify that there is hydrogen coming from the external supply. Check that flow and pressure are turned on at the keyboard. The hydrogen flow rate should be between 1.0 and 5.5 mL/min.
- The bead is not activated. Look through the vent hole on the detector lid to see if the bead is glowing orange. If the bead is not glowing, check that there is enough current reaching the bead. Check the detector background signal. Reduce the bead voltage to zero to establish a reference level, and then look for a sudden sharp increase in output as the bead voltage increases, which indicates that ignition occurred. If 4 V are being supplied to the bead but it is not igniting, the bead is probably burned out. Replace the bead.
- The bead power cable is bad. Contact your Agilent service representative.
- If the upper ceramic insulator is contaminated, a high offset (2 to 15 pA or more) will occur when the bead is off. This directly affects sensitivity. Replace the ceramic insulator.

No baseline; output signal exceeds 8 million

- The electrometer ribbon cable is not attached to the PC board properly. Be sure to turn the GC off before reattaching the cable! If the signal does not drop to a normal level (<3 pA), you need to replace the electrometer. Contact your Agilent service representative.
- The collector is shorted to the detector housing. Check the insulators.

Baseline level is 0.0

- Broken electrometer. Contact your Agilent service representative.

Large positive baseline upset with very slow recovery to original baseline

- The solvent contains significant concentrations of chlorinated hydrocarbon. Create a time table that turns hydrogen off at the time of injection. When the solvent has passed through the detector, restore the hydrogen flow to the previous operating level. The NPD will usually recover rapidly to a stable baseline.

Baseline does not recover after solvent peak

- Create a time table that turns hydrogen off at the time of injection. When the solvent has passed through the detector, restore the hydrogen flow to the previous operating level. The NPD will usually recover rapidly to a stable baseline.

  Add makeup gas at a flow rate of 5 mL/min.

  A large concentration of solvent has extinguished the hydrogen/air plasma. Increase the bead voltage. Run the detector at a higher offset (for example, 40 to 50 pA).

Adjust offset does not function properly (it is either too high or too low by hundreds of pA)

- A flame is burning at the top of the jet. If the hydrogen flow is too high, the flame at the tip of the jet will continue burning. Turn off the hydrogen flow completely, and decrease the flow rate. The hydrogen flow should never be higher than 4.0 mL/min.

Large solvent signal with very small NPD signal

- Check the hydrogen flow rate. If it is too high, a flame could be burning at the tip of the jet. Turn off the hydrogen flow completely, and decrease the flow rate. The hydrogen flow should never be higher than 4.0 mL/min.
- The collector may be contaminated. Change the collector and ceramic insulators.

Peak tailing

- Verify that a good liner and column are being used.
- Some polar compounds tail due to contact with the metal collector. The optional extended jets are recommended.
- Some compounds cause peak tailing, especially those containing phosphorus. The optional black ceramic bead is recommended for phosphorus.

The baseline drifts (upward) significantly during an oven program

- If the oven temperature is increasing dramatically during a run (for example, from 50 to 350°C) a change of between 10 and 15 pA is normal. However, if you suspect that the baseline drift is excessive, heat the inlets and oven to a temperature above 300°C for 60 minutes to eliminate excess baseline drift during oven programs.
- Verify that the detector insulation is not cracked or damaged.

High detector baseline of GC at room temperature

- Moisture in the detector can cause the baseline to be at 10s or even 100s of pA when the detector is at a low (such as room) temperature. Set the detector temperature to 150°C with the detector gases on. The baseline should drop below 1 pA in approximately 10 minutes.

The signal baseline does not fall below 3 pA when the bead voltage is 0

- The ceramic insulators may be dirty. The insulators must be very clean for NPD performance to be satisfactory. Refer to the cleaning procedure on page 590, “Cleaning collector and detector, changing insulators and rings.”

Procedure: Replacing the bead assembly

The bead, which is also referred to as the “source,” is the active part of the NPD. The bead is part of an assembly consisting of a cable that terminates in a connector and a metal hex on which the ceramic bead is mounted.

The NPD bead assembly needs to be removed for replacement or to allow you to access the collector for cleaning.

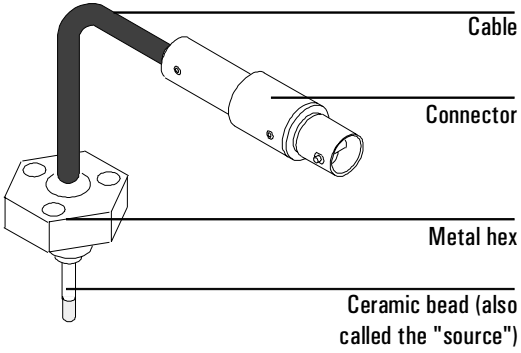


Figure 86. The NPD bead assembly

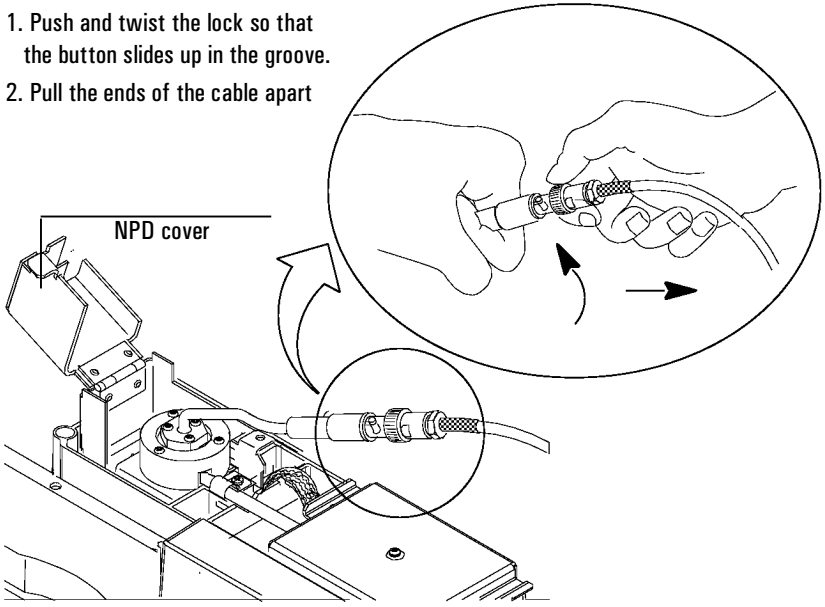
Caution The ceramic bead is delicate. Be careful not to break or crack the bead. When you perform maintenance on the NPD, avoid touching the bead with your fingers, and prevent it from coming in contact with other surfaces.

Caution Be careful! The oven or detector fittings may be hot enough to cause burns.

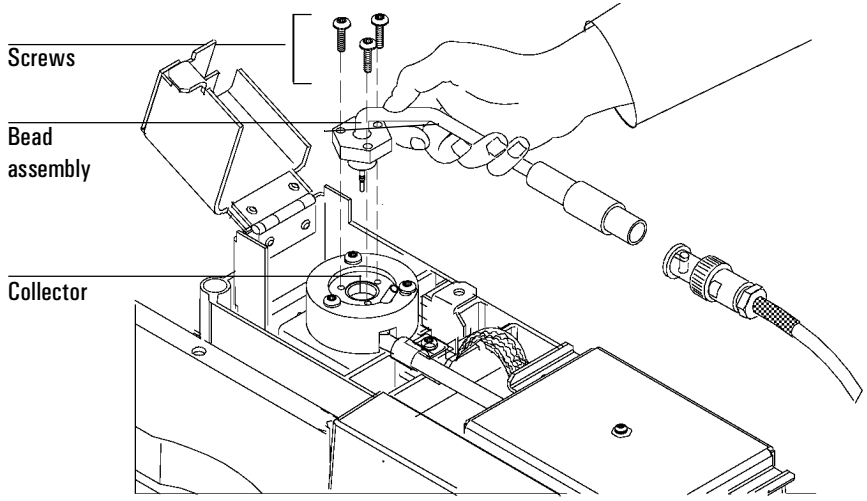
Materials needed:

- T-10 Torx screwdriver
- Cap for the bead

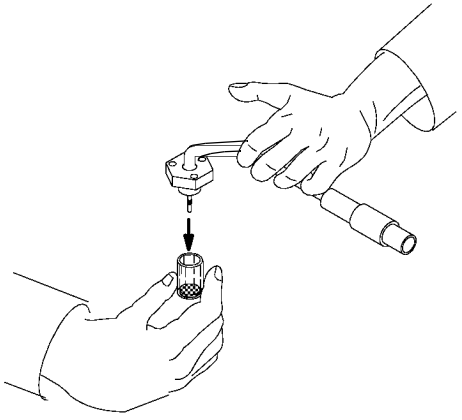
1. Complete the following preliminary steps:
    - Cool the detector to 100°C or lower before changing the bead.
    - Raise the GC top cover and open the NPD cover to cool the detector faster.
    - Turn the detector off. Set the bead voltage to less than 2.0 volts. Leave all gases on.
    - Remove the GC detector top cover and remove the electronics top cover.
2. Disconnect the cable by twisting the ring and pulling the ends apart.



3. Use the Torx screwdriver to remove the three screws on the bead assembly. Grasp the cable gently and lift the bead assembly straight up. Avoid bumping the bead against the sides of the collector.

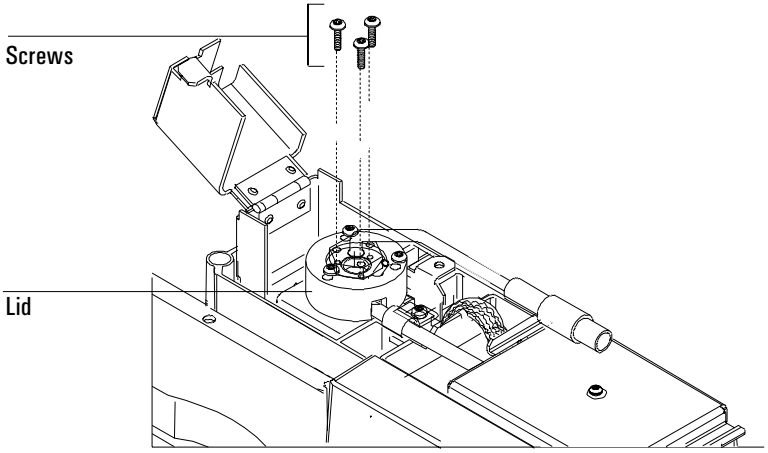


4. Uncap the new bead by pushing the cap off from the cable side. Make sure not to bump the bead on the sides of the cap.

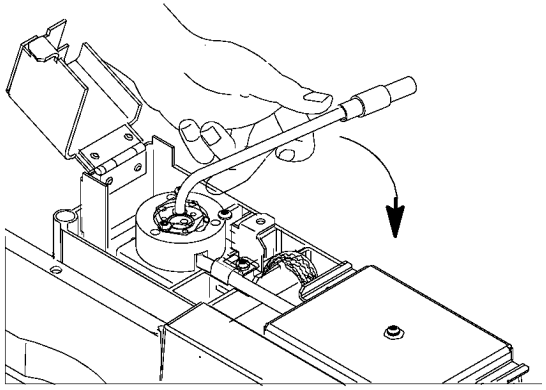


5. Mount the new bead assembly on the NPD lid. Be careful not to bump the bead on the sides of the lid or collector. Replace the three screws. Tighten

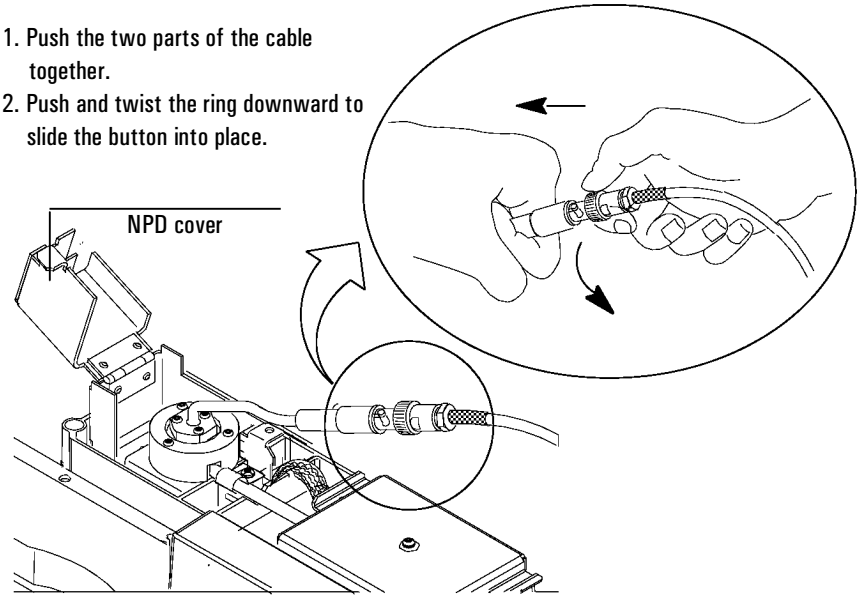
the first screw only finger-tight; tighten the remaining two screws normally and then completely tighten the first screw.



6. Carefully bend the bead assembly cable 90 degrees. You should support the bead as shown below.



7. Reattach the bead assembly power cable to the NPD power cable and twist the ring to lock the connection.



8. Close the NPD cover and the GC detector cover. Replace the electronics top cover. You must close all three covers to get a stable NPD baseline. You can also restore normal operating conditions.

9. Heat the detector to 150°C for about 15 minutes. Then increase the temperature to the operating value (325 to 335°C recommended). Allow 15 minutes for equilibration.

10. Set Equilibration time to 0.0. Either start `Adjust offset` or gradually raise the bead voltage, about 0.01 volts at a time, until the baseline increases to the desired offset.

Procedure: Cleaning detector and collector; changing insulators and rings

Over time, residue from the bead or sample can build up in the collector and cause baseline problems. You should clean the collector after you have changed the bead two or three times.

The ceramic insulators must remain very clean to provide a steady baseline. Always wear gloves when handling the insulators. Clean insulators should provide no more than 1.0 pA, and usually about 0.5 pA, offset with the hydrogen turned off or the bead voltage at 0.

The metal C-rings wear a little with each assembly and disassembly. After several assemblies and disassemblies (five or more), the rings may not seal effectively, causing an erratic baseline. A ceramic insulator and seal kit is available (part no. 5182-9722). Always cool the detector to near-ambient when changing seals and insulators.

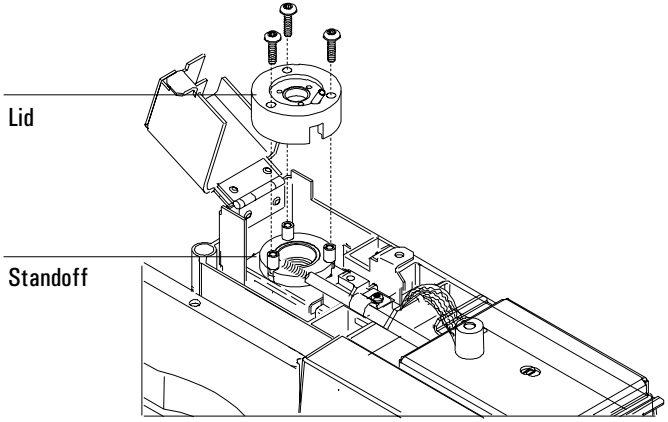
Caution Be careful! The oven or detector fittings may be hot enough to cause burns.

Materials needed:

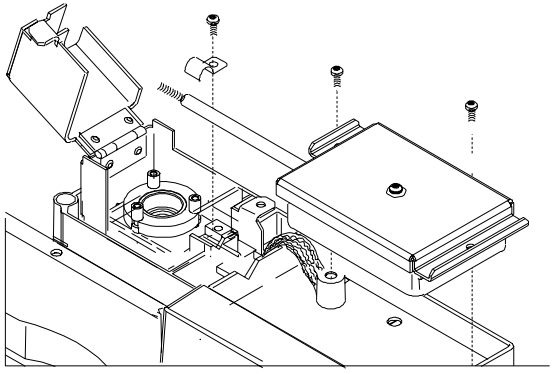
- T-10 and T-20 Torx screwdrivers
- Cap for the bead
- Cotton swabs
- Methanol or acetone
- Compressed air or nitrogen
- Lint-free gloves
- Forceps or tweezers
- New metal rings and ceramic insulators (kit part no. 5182-9722)

1. Complete the following preliminary steps:
   - Cool the detector to 60°C or lower. To cool the detector faster, raise the GC detector cover and open the hinged NPD cover.
   - Turn off the temperature, gases, and bead voltage.
   - Turn off the electrometer; press [Config] [Front Det] or [Config] [Back Det], scroll to `Electrometer` and press [Off].
   - Remove the electronics top cover.
2. Put on the lint-free gloves before touching any of the detector parts.
3. Remove the bead. Refer to the procedure on page 585 for instructions. Cap the bead carefully.

4. Using the T-20 screwdriver, remove the three screws that secure the lid, and then remove the lid. The metal ring and ceramic insulator may be attached to the lid.

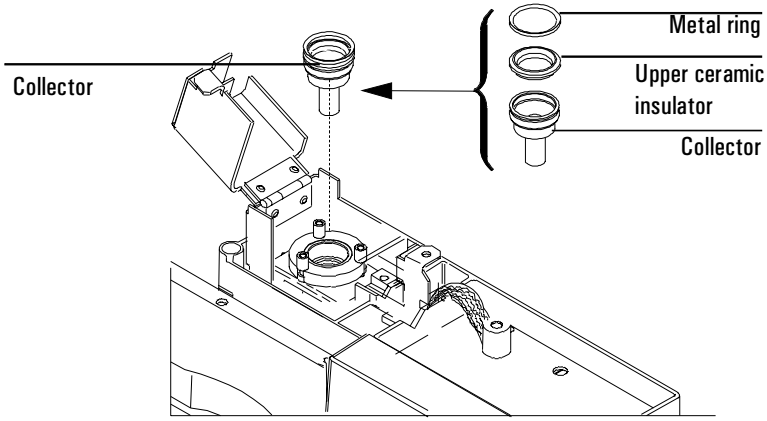


5. Remove the three screws that secure the electrometer and the interconnect. Pull the electrometer away from the detector to free the interconnect. Turn the electrometer to the right to obtain working space.

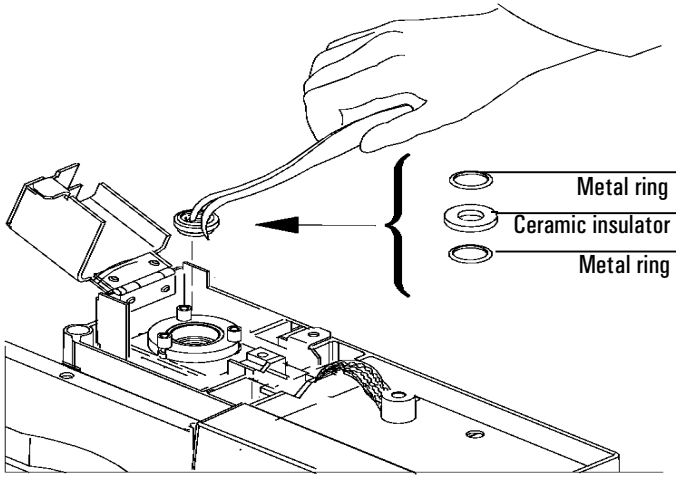


6. Remove the large metal ring and the upper ceramic insulator if they were not attached to the lid. Remove the collector. If you are operating the detector

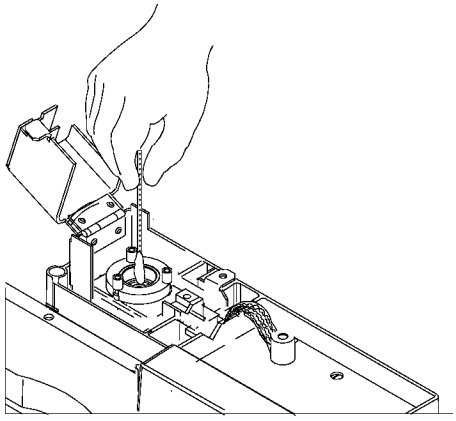
at high temperatures, these parts may stick inside the detector. Push and wiggle them to break the seal.



7. Using the forceps, remove the lower ceramic insulator and the two small metal rings located above and below it. If these parts are stuck together, do not separate them. If they are not stuck, remember which metal ring was on top of the insulator and which was below it! You will need to reassemble the pieces in the same orientation.



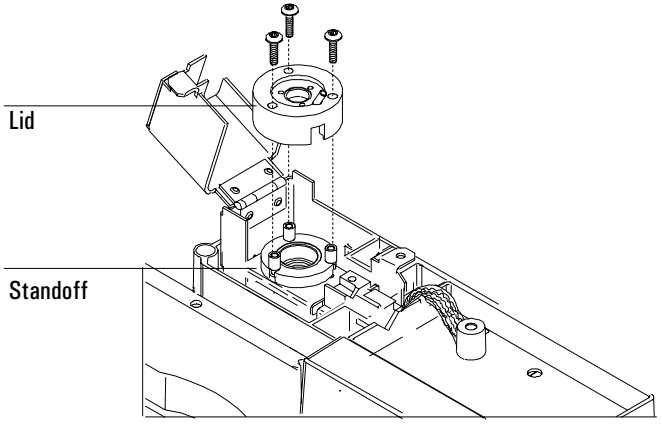
8. Use a cotton swab wetted with solvent to clean the residue from the inside of the collector and around the “cup.” Also swab the detector base around the jet with a swab.



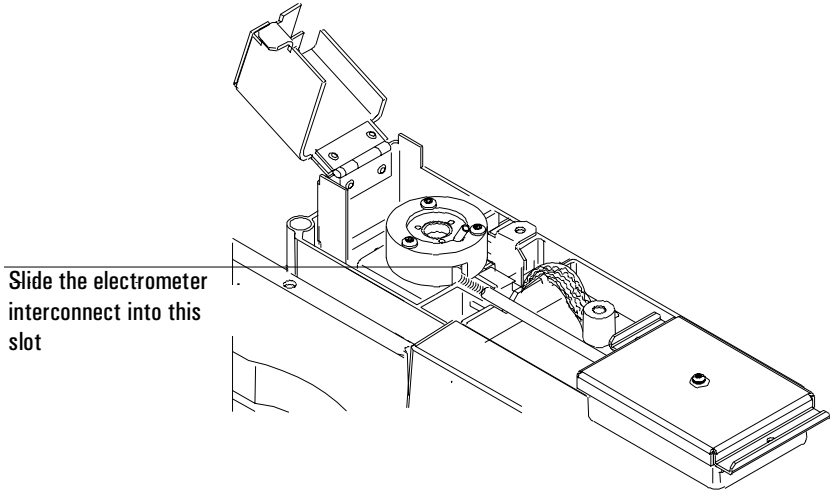
If the collector or the upper ceramic insulator are really dirty, cleaning may not help. Replace with new parts.

9. Reinstall the old or insert the new bottom metal ring, the lower ceramic insulator, and the second metal ring. Install the clean (or new) collector. Reinstall the old or insert the new upper ceramic insulator and large metal ring on top of the collector.

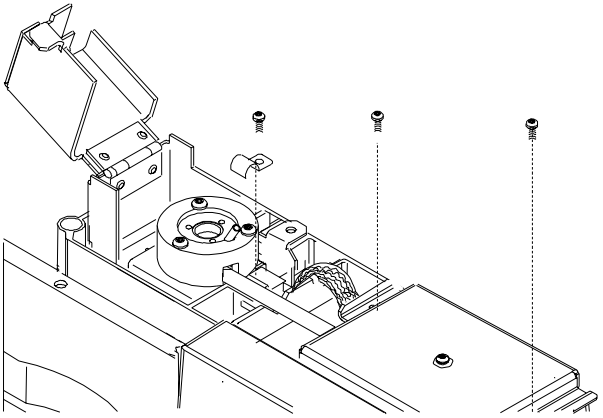
10. Replace the lid, making sure that the three standoffs are in their slots. Hold the lid flat while each of the three screws are tightened until they touch the lid. Tighten each one-half a turn at a time until tight.



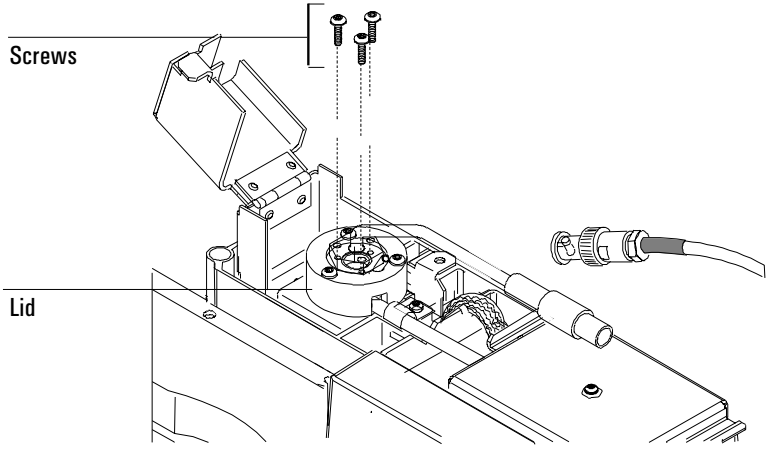
11. Slide the electrometer interconnect into the slot on the lid. Lower the electrometer into the mounting tray.



12. Replace the bracket, and replace and tighten the three screws.



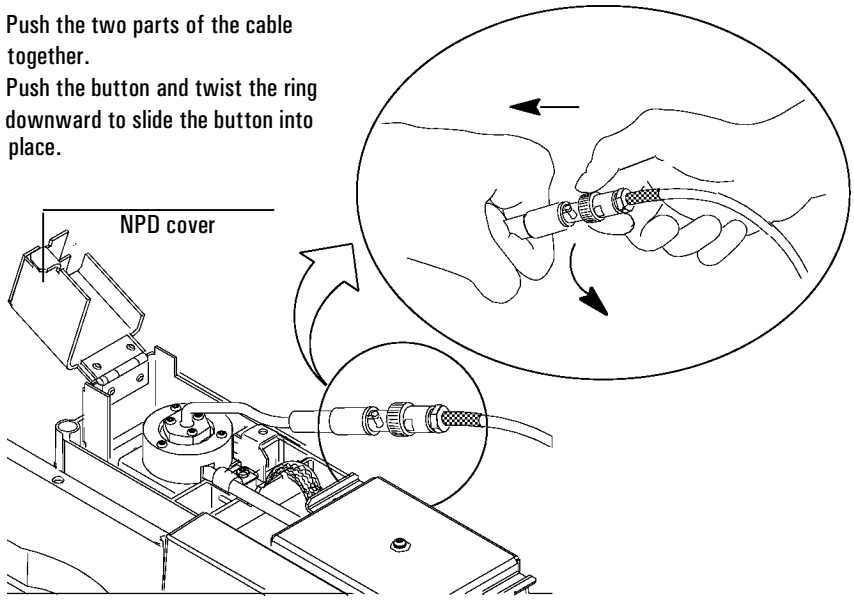
13. Uncap the bead and replace the bead. Replace the three screws. Tighten the first screw only to snugness. Tighten the other screws completely, and then completely tighten the first screw.



14. Reattach the bead assembly cable to the NPD power cable and twist the ring to lock the connection. Close the NPD cover and the GC detector cover and

replace the electronics top cover. You can restore normal operating conditions.

1. Push the two parts of the cable together.
2. Push the button and twist the ring downward to slide the button into place.



After reassembling the detector, you should check its operation. Turn on the gases, and then turn the bead voltage on to restore detector operation. Check that the offset reading is appropriate for your detector. If the values are not normal, the spring on the electrometer may not be contacting the detector correctly, there may be a leak at the column connection, or the detector may not have been reassembled correctly.

Replacing or cleaning the jet

Because there is no flame in the NPD, the jet does not collect silica and soot as does the FID jet. Although you can clean the jet, it is usually more practical to simply replace dirty jets with new ones. If you do clean the jet, use the cleaning wire (part no. 18765-20070), taking care not to damage the inside of the jet. You can also use a sonicator bath to clean the jet.

Table 66 lists the NPD jets.

Table 66. NPD Jets

Type	Part no.	Use with
Standard jet	G1531-80560	Capillary-optimized NPD
Extended jet (optional)	G1534-80580	Capillary-optimized NPD
Extended jet (optional)	G1534-80590	Adaptable NPD

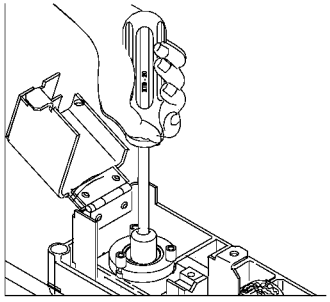
There are four steps involved in cleaning the jet: removing the jet, inspecting it for damage or wear, cleaning the jet (optional), and replacing the jet and reassembling the detector.

Procedure: Removing and inspecting the jet

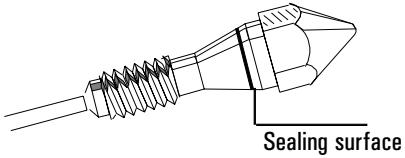
Materials needed:

- T-10 and T-20 Torx screwdrivers
- 1/4-inch hex driver
- Cap for the bead
- Lint-free gloves
- Forceps or tweezers
- ESD wrist strap

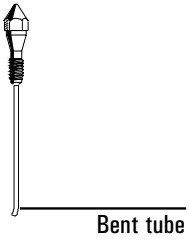
1. Complete the following preliminary steps:
   - Raise the top cover and the NPD cover. Cool the detector to 60°C or lower. Turn off the inlet gases.
   - Turn off the temperature, gases, and bead voltage.
   - Turn off the electrometer; press [Config] [Front Det] or [Config] [Back Det], scroll to `Electrometer` and press [Off].
   - Cool the oven to room temperature. Remove the column from the detector end and cap the detector's column connection.
   - Open the GC detector cover and remove the electronics top cover.
2. Remove the collector, ceramic insulators and metal rings. Refer to the procedure starting on page 590.
3. Using the nut driver, loosen the jet. Pull the jet straight out of the detector. You may need to use the forceps to remove it.



4. Inspect the jet sealing surface for scratches. You should see a small ring around the sealing surface; any other scratches, however, are unacceptable.



5. Inspect the jet tube to be sure it is not bent or crimped.



6. Inspect the jet for contamination by holding it up to a light and looking through its bore. If no contamination is present, the tube will be clear.

Caution The adaptable NPD extended jet is longer than the capillary-optimized NPD extended jet and should never be installed in a capillary-optimized detector.

Procedure: Cleaning the jet

It is often more convenient to replace dirty jets with new ones than to clean them, especially jets that have been badly contaminated.

Caution If you choose to clean a jet, be careful when using a cleaning wire. Be sure not to scratch the jet, because doing so will ruin it. You may want to skip the wire cleaning procedure and use the aqueous bath only.

Materials needed:

- Small ultrasonic cleaning bath
- Aqueous detergent
- GC-grade methanol in a Teflon wash bottle
- Flame detector cleaning kit (part no. 9301-0985)
- Dry, filtered, compressed air or nitrogen

1. Run a cleaning wire through the jet. Run it back and forth a few times until it moves smoothly. Be careful not to scratch the jet.
2. Aqueous cleaning procedure:
   a. Fill the ultrasonic cleaning bath with aqueous detergent, and place the jet in the bath. Sonicate for 5 minutes.
   b. Use a jet reamer to clean the inside of the jet.
   c. Sonicate again for 5 minutes.

   From this point on, handle the parts only with forceps!
   a. Remove the jet from the bath and rinse it thoroughly first with hot tap water and then with a small amount of methanol.
   b. Blow the jet dry with a burst of compressed air or nitrogen, and then place the jet on a paper towel to air dry.

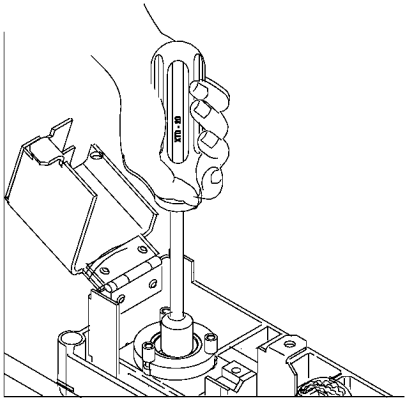
Procedure: Replacing the jet and reassembling the detector

Materials needed:

- T-10 and T-20 Torx screwdrivers
- Cap for the bead
- ESD wrist strap
- Lint-free gloves

Caution The adaptable NPD extended jet is longer than the capillary-optimized NPD extended jet and should never be installed in a capillary-optimized detector.

1. Place the jet in the detector body, and tighten it to snugness with the hex driver. Do not overtighten the jet.



2. Reassemble the detector. Refer to the procedure starting on page 594.

25 The Micro-Cell Electron Capture Detector

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The Micro-Cell Electron Capture Detector

Regulatory and Safety Information

This chapter describes the micro-cell detector (μ-ECD).

The μ-ECD contains a cell plated with ^{63}Ni, a radioactive isotope. The ^{63}Ni releases β particles that collide with carrier gas molecules to produce low-energy electrons—each β particle produces approximately 100 electrons. The free electrons produce a small current—called the *reference* or *standing current*—that is collected and measured in a pulsed circuit.

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

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

The ^{63}Ni isotope

The radioactive isotope used in the cell is ^{63}Ni. It is plated onto the inner surface of the cell body and is solid at temperatures used in chromatography. Some other properties are listed in Table 67.

Table 67. Properties of ^{63}Ni

Half–life:	101.1 years
Emission:	65.87 keV max., beta radiation
Melting point:	1453°C
Dimensions of the active part of the μ-ECD:	Inside diameter: 6 mm Height: 4.2 mm
Total activity (μ-ECD cell):	555 MBq (15 millicuries) maximum

ECD licenses

Customers in the United states can purchase a µ-ECD under either a General License or a Specific License. Customers outside the United States should contact their local Agilent sales office for information.

Specific License

Specific license µ-ECDs require you to obtain a Materials License from the Nuclear Regulatory Commission (NRC) or the local state agency, permitting you to possess the amount and kind of radioisotope used in the detector. You can typically ship, sell, or transfer the µ-ECD to other Specific Licensees. If the license permits, you may also open the µ-ECD for cleaning.

General License

General License ECDs do not require a Materials License. You become a General Licensee automatically when you purchase a µ-ECD directly from Agilent Technologies . Some states may require that you register the µ-ECD with a state agency.

Certain restrictions apply to General Licenses:

1. Owners may not open the μ-ECD cell.
2. Owners shall not modify the cell in any manner.
3. Owners shall not use any solvent, including water, to internally clean the cell.
4. Owners shall not interfere with or attempt to defeat the overheat circuitry that may be supplied with the μ-ECD.
5. Owners shall not transfer the μ-ECD to another person or another location except as described in the applicable Regulations.
6. Owners must perform a radioactive leak test at least every 6 months.
7. Owners must maintain records as required by your local Agency (the NRC or, in certain states, a state agency).
8. Owners must notify the Agency in case of incidents or failures that might lead to a hazardous condition.

Additional information is available in the publication “Information for General Licensees,” part no. 5961-5664.

μ-ECD warnings

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

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

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

- After turning off the main power and allowing the instrument to cool, cap the detector inlet and exhaust vent openings. Wear disposable plastic gloves and observe normal laboratory safety precautions.
- Return the cell for exchange, following directions included with the License Verification Form (part no. 19233-90750).
- Include a letter stating the condition of abuse.

It is unlikely, even in this very unusual situation, that radioactive material will escape the cell. However, permanent damage to the ^{63}Ni plating within the cell is possible, and therefore, the cell must be returned for exchange.

WARNING Do not use solvents to clean the µ-ECD.

WARNING You may not open the µ-ECD cell unless authorized to do so by your local nuclear regulatory agency. Do not disturb the four socket-head bolts. These hold the cell halves together. Removing or disturbing them is a violation of the terms of the General License and could create a safety hazard.

Safety precautions when handling µ-ECDs

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

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

General Information

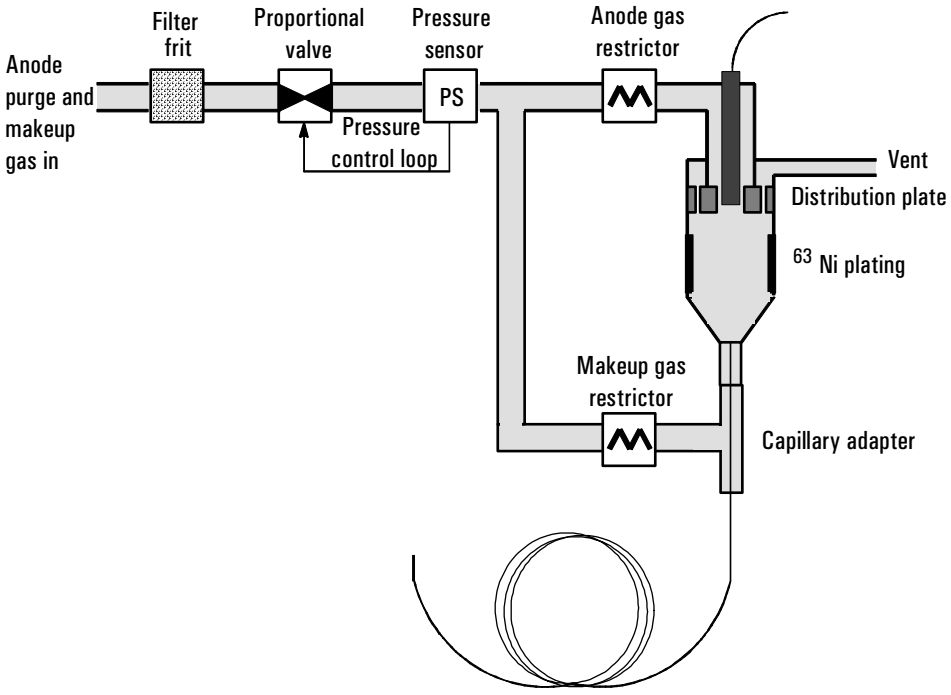


Figure 87. μ-ECD pneumatics

Linearity

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

Detector gas

The μ-ECD operates with either nitrogen or argon/methane as the makeup and anode gas.

Because of the high detector sensitivity, carrier and makeup gas must be dry and oxygen-free. Moisture, chemical, and oxygen traps in good condition should be installed in carrier and makeup gas supply lines.

Temperature

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

Electrometer

The Configure Detector control table contains an `On/Off` setpoint for the `Electrometer`. Keep the electrometer on all the time when operating your detector.

Operating the μ-ECD

If you intend to use the analog output from the μ-ECD, you must set the output Range to 10. This is done by pressing

[SIG 1] [RANGE] [10] [ENTER]

Use the information in Table 68 when selecting temperatures and flows. Maximum source pressure must not exceed 100 psi. Use the maximum source pressure to achieve maximum makeup flow rate.

Table 68. Operating Parameters

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

Notes

1. If the carrier gas type is different from the makeup gas type, the makeup gas flow rate must be at least three times the carrier gas flow rate.
2. μ-ECD sensitivity can be increased by reducing the makeup gas flow rate.
3. μ-ECD chromatographic speed (for fast peaks) can be increased by increasing the makeup gas flow rate.

Procedure: Operating the μ-ECD

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

1. Press [Front Det] or [Back Det] to open the μ-ECD control table.
2. Set the detector temperature. To keep the μ-ECD cell clean, this temperature must be higher than the oven temperature.

Caution Detector electronics depend on the correct gas configuration.

Short-cut procedure: *(assumes correct setpoints are stored)*

1. Open detector control table.
2. Turn temperature On.
3. Turn makeup gas On, if needed.
4. Press [Det Control] and check Output.

3. Verify that the makeup gas type is the same as that plumbed to your instrument. The gas type is in parentheses next to the `Mkup` line on the control table. Change the gas type, if necessary.
4. Enter a value for the makeup gas.

   If you are using ***packed columns***, turn off the makeup gas.

   If your ***capillary column*** is *defined*, choose a new flow mode, if desired, and set the makeup or combined gas flow.

   If your capillary column is *not defined*, only constant makeup flow is available. Enter a makeup gas flow.

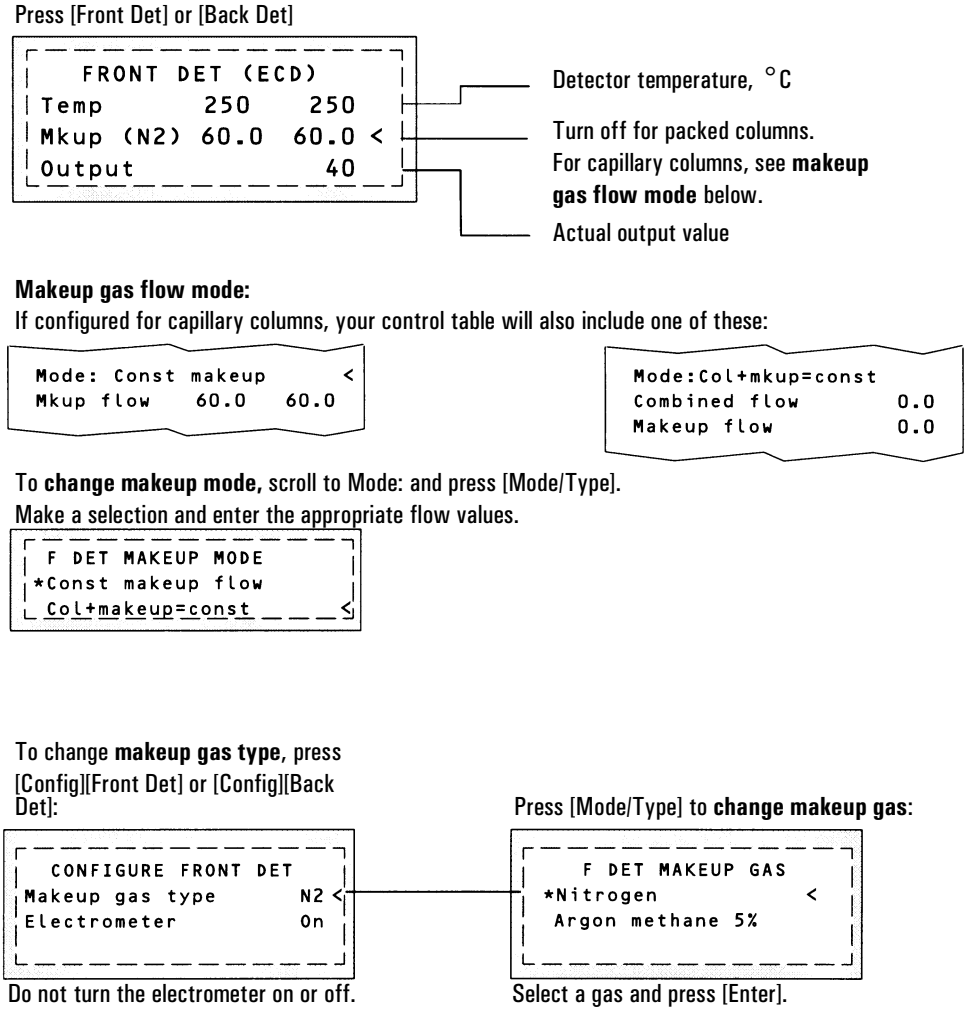


Figure 88. μ-ECD control table

Checkout Conditions and Chromatogram

This section contains a typical example of a test sample chromatogram. It may be used as a general guide to instrument performance.

Note that injection volumes listed with operating conditions do not necessarily indicate total absolute volume injected. Volume given is simply the graduation (plunger position) read from a standard 10 μL syringe. For a heated inlet, actual sample volume injected will also include an additional 0.4-0.7 μL, the volume of sample volatilized from inside the syringe needle. For the dedicated, on-column inlet (unheated), the syringe plunger position more accurately reflects the true injected volume.

Also note that the following procedure and results are intended only to provide evidence of a properly functioning inlet and/or detector system; they are not necessarily suitable to test a given system against its specification limits.

μ-ECD checkout conditions

Column and sample

Type	HP-5 30m × 0.32mm × 0.25 μm PN 19091J-413
Sample	ECD Checkout 18713-60040
Injection volume	1 μL

Inlet

Temperature	200° C Purged packed
	250°C Split/splitless
	Oven Track Cool On-Column
	80°C PTV (see below)
Inlet pressure	25 psi (Constant pressure for EPC inlets, helium)

Split/Splitless

Mode	Splitless
Purge flow	60 mL/min
Purge time	0.75 min

Inlet, continued

PTV

Mode	Splitless
Inlet temperature	80°C
Initial time	0.1 min
Rate 1	720°C/min
Final temp 1	350°C
Final time 1	2 min
Rate 2	100°C/min
Final temp 2	250°C
Final time 2	0 min
Inlet pressure	25 psi (Constant pressure for EPC inlets)
Purge time	0.75 min
Purge flow	60 mL/min

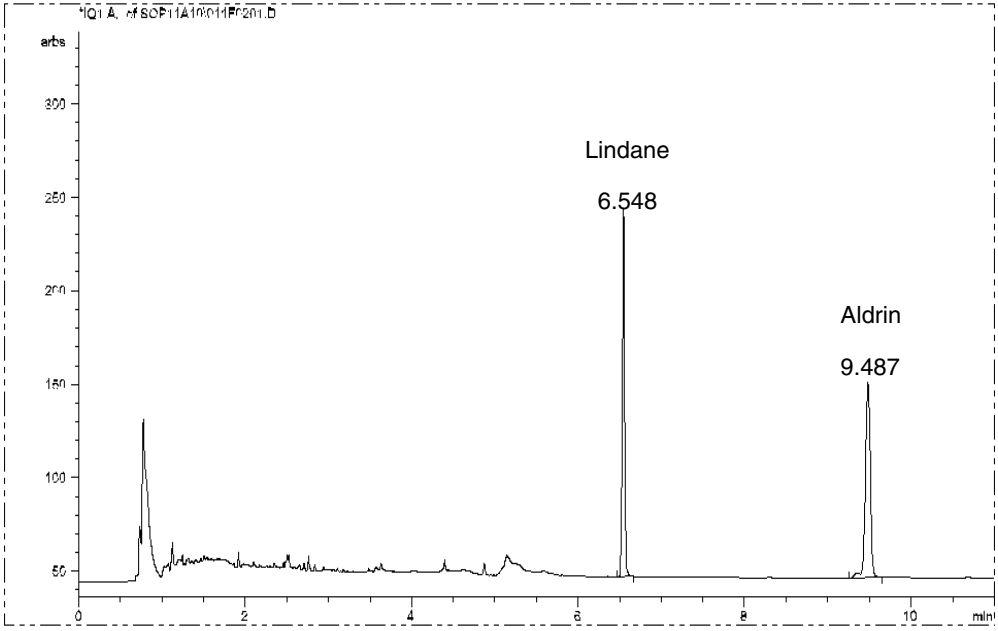
Detector

Temperature	300° C
Anode purge, nitrogen	60 mL/min
Makeup, nitrogen	25 ± 2 mL/min
Offset	Should be < 1000 display counts

Oven

Initial temp	80° C
Initial time	0 min
Rate 1	15° C/min
Final temp	180° C
Final time	10 min

Typical µ-ECD checkout chromatogram



Your retention times will differ but peaks should resemble the example.

Maintaining the Detector

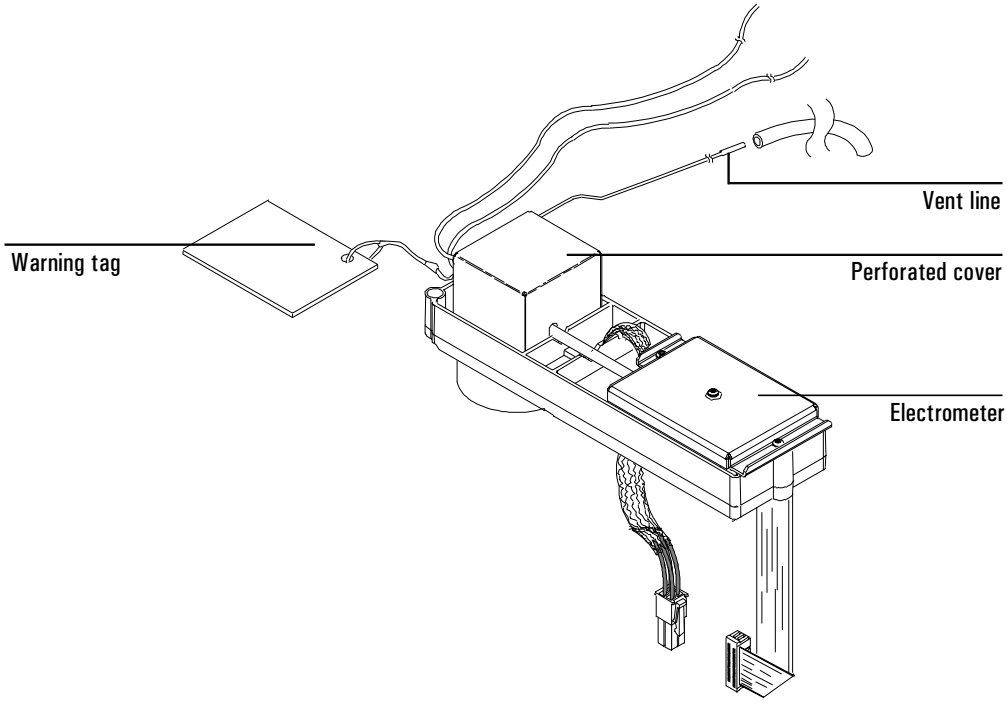


Figure 89. The μ-ECD

Correcting performance problems

Performance problems, such as an output reading that is too high or too low or unsatisfactory chromatographic results (for example, a noisy baseline), can be caused by leaks or deposits in the detector or other part of the chromatographic system. To determine the location of the problem, you need to perform a series of tests.

Before testing the detector, consider the nature of the problem. If you have recently made a change to the GC system and now see an elevated output level, there is a good chance that the change has either introduced contaminants or caused a leak in the system. For example, if you recently switched gas supplies, the new gas may contain impurities. Or if you recently installed a new column, there could be a leak at the detector fitting.

If the output value or noise level has been increasing gradually, the cause is probably a slow build-up of deposits. The detector may contain contaminants from column bleed or a trap may be saturated. If the change has been gradual and if you have not modified the GC system recently, you can probably start by checking for contamination. *Note: Contamination in this procedure refers to non-radioactive deposits from such things as column bleed or dirty samples!*

1. Make sure the detector is operating under normal conditions and that at least 2 hours have lapsed since the last run.

   Check the output value in the detector control table. If it differs considerably from the normal output level—either too high or too low—you should continue with this procedure to identify the cause of the abnormal reading.

2. Use an electronic leak detector to check for leaks at the inlet and detector and the column fittings. Correct leaks and then check the output level. If it is still abnormal, continue to step 3.

3. The detector itself is not a likely source of leaks, so you should leak test the inlet if the output reading is still abnormal. See the maintenance material for your inlet in "The Split/Splitless Inlet", "The Purged Packed Inlet", "The Cool On-Column Inlet", "The Programmable Temperature Vaporization Inlet", "The Volatiles Interface".

If the inlet is not leaking, go to step 4 to check for leaks in the detector.

If the inlet is leaking, correct the leaks and check the output. If it is still abnormal, the detector also may be leaking. Go to step 4.

4. Follow the leak test for the detector later in this document.

   If the detector is not leaking, the cause of the problem is contamination. Go to step 5.

   If the detector is leaking, correct the leaks, and then recheck the output. If it is still abnormal, go to step 5.

5. Check for contamination:

   a. Remove the column and plug the detector connection with the cap (part no. 19234-20650) and cap nut (part no. 19234-20570).

   b. Run the detector at your normal operating conditions but with only makeup gas flowing through it. Monitor the output. If it is normal for your detector, then the contamination is from another part of the GC system. Go on to step 6.

   c. If the output is abnormal, then the detector is contaminated. Perform a thermal bake out to decontaminate the detector. The procedure is described later in "Thermal cleaning".

6. One part at a time, check the rest of the GC system for contamination by making the following changes and monitoring the output:

   - Replace the column with an empty column and compare the output readings.
   - Switch to a different inlet (if possible), and compare the output.
   - Switch to a different source of gas and compare the output.
   - Replace the traps; compare the output.

Checking for gas leaks

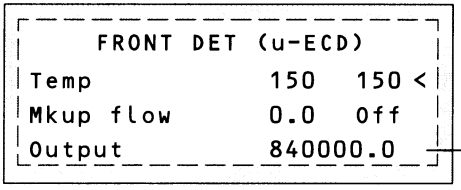
The detector is an unlikely leak source. If you suspect that there is a leak in your GC system and have checked the gas plumbing to the GC, the inlet, and the column inlet and detector connections without finding it, follow this procedure to test the detector.

The oven and inlet should be at their normal operating temperatures.

Materials needed:

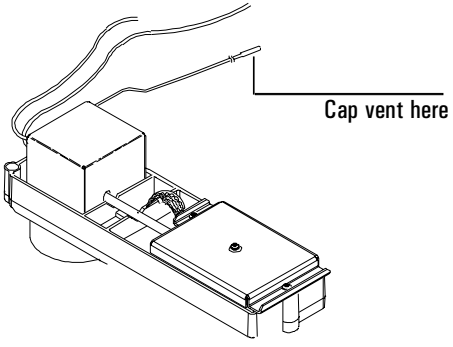
- A vent plug (part no. 5060-9055)
- An electronic leak detector capable of detecting your carrier gas

1. Turn off the inlet pressure. Allow some time to purge the system of the gas.
2. Turn off the makeup gas flow.



When there is no flow, the output will be at its maximum, which is approximately 840,000 for both argon/methane and nitrogen

3. Cap the detector exhaust vent with the vent plug.



4. Set pressure at the inlet to 15 psi (103 kPa). Monitor the system pressure from the inlet. Allow time for the system to become fully pressurized (at least 1 minute). When the system is fully pressurized turn off the pressure or the gas.

   Monitor the pressure for 10 to 15 minutes. If the pressure stays stable or drops only by 0.2 or 0.3 psi/min, you can consider the detector leak-free. If pressure drops, you have a leak. Continue to step 5.

5. Use the electronic leak detector to check for leaks at the column fitting and plugged vent. If you find leaks, tighten the fittings, and repeat the leak test.

   If the other system components are leak-free, then the detector may be leaking. The detector cannot be disassembled without special license from the Nuclear Regulatory Commission or Agreement State Licensing Agency (USA only). Contact your Agilent service representative for more information.

Thermal cleaning

If your baseline is noisy or the output value is abnormally high and you have determined that these problems are not being caused by leaks in the GC system, you may have contamination in the detector from column bleed. To remove contamination, you should perform a thermal cleaning (also called “bake-out”) of the detector.

WARNING Detector disassembly and/or cleaning procedures other than thermal should be performed only by personnel trained and licensed appropriately to handle radioactive materials. Trace amounts of radioactive ^{63}Ni may be removed during other procedures, causing possible hazardous exposure to β- and x-radiation.

WARNING To prevent possible hazardous contamination of the area with radioactive material, the detector exhaust vent always must be connected to a fume hood, or otherwise vented in compliance with the latest revision of Title 10, CFR, Part 20, or with state regulations with which the nuclear regulatory commission has entered into an agreement (USA only). For other countries, consult with the appropriate agency for equivalent requirements.

Materials needed:

- Cap for the detector connection (part no. 19234-20650)
- The nut to connect the cap (part no. 19234-20570)

1. With the detector and oven at normal operating temperatures, press [Front Det] or [Back Det] to open the control table. Note the value of Output for later comparison.
2. Turn the anode purge and the makeup gas flow off.
3. Remove the column from the detector. Make sure to cap the unconnected end. Install the detector cap and nut into the column detector fitting to plug the connection.

4. Enter the following values:
   - temperature = 350 to 375°C
   - makeup gas = 60 mL/min.
5. Set the oven temperature to 250°C.
6. Allow thermal cleaning to continue for several hours and then cool the system to normal operating temperatures.
7. Check the µ-ECD output value on the control table. It should be lower than the first reading. If it is not, contact your Agilent service representative.

Performing a wipe test (radioactivity leak test)

Electron capture detectors must be tested for radioactive leakage at least every 6 months. Records of tests and results must be maintained for possible inspection by the Nuclear Regulatory Commission and/or responsible state agency. More frequent tests may be conducted when necessary.

The procedure used is the **wipe test**. A wipe test kit is supplied with each new detector. Refer to the information card supplied in the Wipe Test Kit for instructions on performing the test.

26 The Flame Photometric Detector

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The Flame Photometric Detector (FPD)

General Information

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

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

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

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

Linearity

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

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

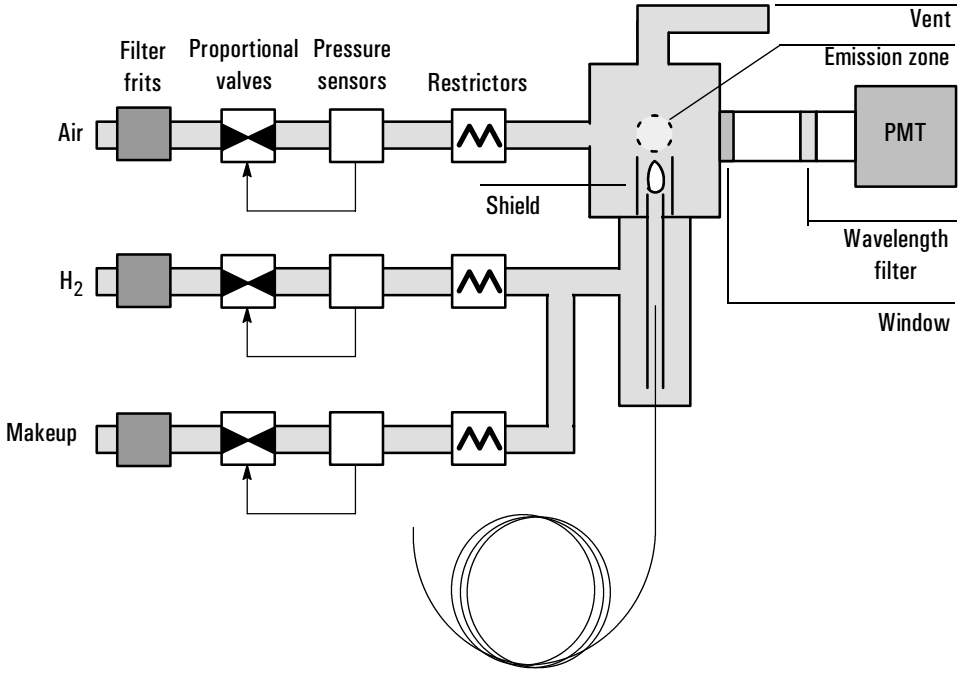


Figure 90. Schematic of a flame photometric detector

Quenching effects

Hydrocarbon quenching occurs when a high concentration of carbon dioxide from a hydrocarbon peak is in the flame at the same time as the sulfur species. Part of the light emitted by the sulfur species is absorbed by some CO_2 species.

Self-quenching occurs at high concentrations of the heteroatom species. Some other ground state (unactivated) species reabsorbs the emitted photon, preventing it from reaching the PMT.

These effects are reduced by good chromatographic practices. The column should provide good separation of the compounds, those that contain sulfur or phosphorus as well as those that do not but may absorb light. A careful, multilevel calibration is well worth the investment!

Detector and gas cleanliness must be maintained to have consistent responses. Since most sulfur and phosphorus compounds contain chemically active sites, the injection and column systems must be kept very clean.

PMT saturation

The photomultiplier tube may saturate if light intensity is too high. When this happens, increasing concentration produces little or no increase in signal and peak tops are rounded or flattened. Dilute the sample to correct the problem.

Optical filters

The filters are marked on the edge with the transmission wavelength. Each filter has a mirrored side—which must face the flame when installed—and a colored surface.

The sulfur filter is blue/purple and transmits at 393 nanometers.

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

Fused silica liner

The FPD uses an inert fused silica insert liner in the transfer line. This allows fused silica columns up to 530 µm ID to run right to the base of the flame, minimizing sample tailing or loss on chemically active sites. The liner is also compatible with standard packed columns.

Conditions that prevent the detector from operating

- Temperature set below 120°C
- Air or hydrogen flow set at `Off` or set at `0.0`
- Ignition failure

Detector shutdown

If a critical detector gas is shut down due to a pneumatics or ignition failure, your detector shuts down. This turns off everything except the detector temperature and makeup gas flow.

Compatibility requirements

If a single wavelength FPD is to be used with an Agilent ChemStation, the ChemStation must be version 4.02 or higher.

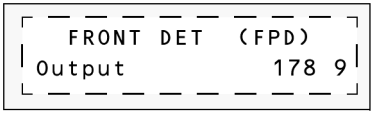
If a dual wavelength FPD is to be used with an Agilent ChemStation, the ChemStation must be version 5.01 or higher.

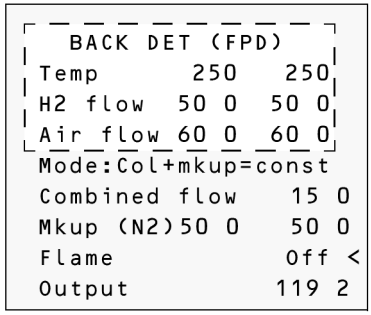
The dual wavelength FPD

This is a single burner module with two PMT housings, one with a sulfur filter and the other with a phosphorus filter. Because the optimum gas flows for these elements are quite different, performance of this detector is a compromise.

The detector mounts in the back position and is heated by the Back Det and AUX 2 heaters. The AUX 2 setpoint is automatically set by the Back Det setpoint.

Two signal channels and two electrometer boards are used, one for each PMT. The Back Det control table runs the detector, while the Front Det operates in a special "signal only" mode. Typical tables for a dual wavelength FPD are:





If a heated zone is assigned to the Front Det position, an "F det type mismatch" will be declared. To override this, press [Config], scroll to the `Instrument` line and press [Enter]. Scroll to the `F det` line, press [Mode/Type], and select `Sig only FPD`.

Using the Detector

Detector temperature considerations

The FPD flame produces considerable water vapor. The detector must be operated above 120°C to prevent condensation.

Unnecessarily high temperatures can cause thermal decomposition of many thermally labile phosphorus and sulfur compounds.

Detector temperature can have a significant effect on sulfur sensitivity. If analyzing compounds with high boiling points, the detector temperature should be set to 25°C above the final oven temperature—if allowed by the temperature limit of 250°C.

Heater configuration

The FPD burner module has two heated zones, one for the detector body and one for the transfer line.

A single wavelength FPD can be mounted in either the front or back position. In the front position, it uses the Front Det and AUX 1 heaters. In the back position, it uses the Back Det and AUX 2 heaters. A second detector—possibly another FPD—can be mounted in the unused position.

A dual wavelength FPD—simultaneous detection of sulfur and phosphorus—must be mounted in the back position, where it uses the Back Det and AUX 2 heaters. A second detector cannot be mounted.

The software automatically sets the AUX heater to the same setpoint as the Det heater. You do not have to contend with two separate entries.

Lit offset

Lit offset is the expected difference between the FPD output with the flame lit and the output with the flame off. It is used to determine whether an attempted ignition has succeeded and to detect a flame-out condition.

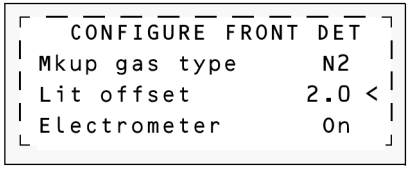
If the output with the flame on minus the output with the flame off is greater than `Lit offset`, the flame is considered lit.

The default setting for `Lit offset` is 2.0 picoamps. This is a good working value for all but very clean gases and systems. You may want to change this setpoint if:

- Your detector is attempting to reignite when the flame is still on, thus producing a shutdown.
- Your detector is not trying to reignite when the flame is out.

Procedure: Changing the Lit offset setpoint

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



2. Scroll to `Lit offset` and enter a number.
   The default is 2.0 pA.
   Enter 0 to disable the automatic reignite function.
   The setpoint range is 0 to 99.9 pA.

Flame ignition sequence

When either of the flame ignition methods on the next page is used, the FPD automatically performs this sequence:

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

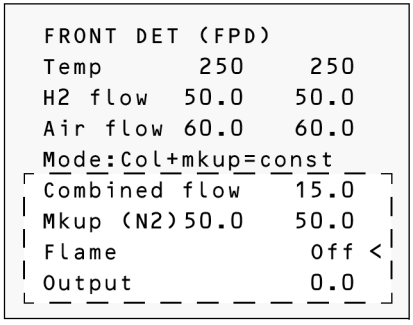
For this process to work, there must be enough air pressure to the pneumatics module to provide 200 mL/min flow. We recommend a supply pressure of 90 psi.

Lighting the flame

Manual

To start the flame ignition sequence:

Press [Front Det] or [Back Det]



Scroll to Flame and press [On]

Automatic

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

Electrometer on/off

The Configure Detector control table contains an Electrometer On/Off setpoint.

On	High voltage and signal processing circuits are on. **If the photomultiplier tube is exposed to room light with the electrometer on, the tube will be destroyed.**
Off	High voltage and signal processing circuits are off. In this condition, it is safe to expose the photomultiplier tube to room light.

Caution Always turn the electrometer off before removing the PMT housing to avoid destroying the tube.

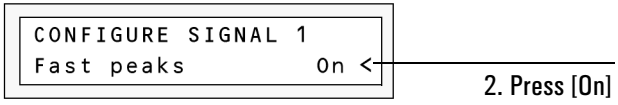
Electrometer data rates

Analog output for the FPD can be presented at either of two speeds. The faster speed allows minimum peak widths of 0.004 minutes, while the standard speed allows peak widths of 0.01 minutes.

Procedure: Using fast peaks

If you are using the *fast peaks* feature, your integrator must be fast enough to process the data coming from the GC. It is recommended that your integrator bandwidth be at least 15 Hz. To use fast peaks:

1. Press [Config][Signal 1] or [Config][Signal 2]



The fast peaks feature does not apply to digital output.

Operating the FPD

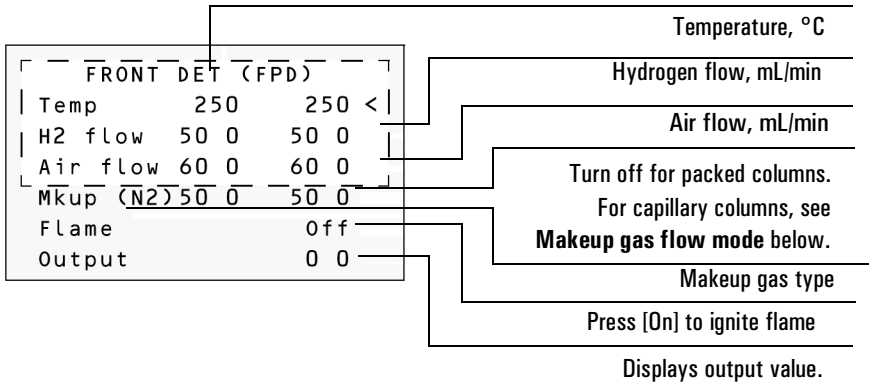
Table 69 gives the flows for the maximum sensitivity FPD flame, which is hydrogen-rich and oxygen-poor. It is difficult to light the flame with these flows, particularly in the sulfur mode. Helium, used as carrier or makeup gas, may cool the detector gases below the ignition temperature. We recommend using nitrogen rather than helium.

Table 69. Recommended Temperature and Flow

	Sulfur mode flows mL/min	Phosphorus mode flows mL/min
Carrier (hydrogen, helium, nitrogen, argon)		
Packed columns	10 to 60	10 to 60
Capillary columns	1 to 5	1 to 5
Detector gases		
Hydrogen	50	150
Air	60	110
Carrier + makeup	60	60
Supply pressure Air supply pressure: at least 90 psi for the ignition sequence. All others: adequate to achieve desired flows.		
Detector temperature Below 120°C, flame will not light. Set temperature about 25°C higher than highest oven temperature—limit is 250°C.		
Lit offset [Config] [Front Det] **or** [Back Det] If the detector output (with the flame on) minus the output (with the flame off) falls below this value, the FPD attempts to re-ignite twice. If output does not increase by at least this much, the detector shuts down. The recommended setting is 2.0 pA. A setting of 0 or [Off] disables autoignition.		

If the flame will not light with the sulfur mode flows shown, change to the phosphorus mode values. After the flame lights, gradually reduce the flows toward the sulfur mode values. Some experimentation will be required to find flows for your particular detector.

Press [Front Det] or [Back Det].



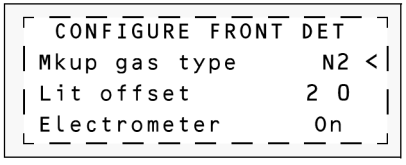
Makeup gas flow mode: If column dimensions are specified, the control table will also include one of these sets.

```
Mode:Const makeup   <
Mkup flow 0.0     Off
```

```
Mode:Col+mkup=const
Combined flow     0.0
Makeup flow       0.0
```

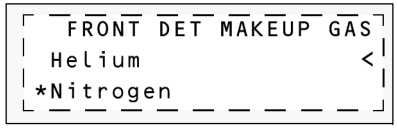
To **change makeup mode**, scroll to Mode: and press [Mode/Type]. Make a selection and enter the appropriate flow values.

To view **makeup gas** or change **Lit offset**, press [Config][Front Det] or [Config][Back Det]:



It is not necessary to turn the electrometer on or off unless you are performing a maintenance procedure.

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



Select the appropriate gas.

Figure 91. FPD control table

Procedure: Using the FPD

Verify that all detector gases are connected, a column is installed, and the system is free of leaks. Check the oven temperature, inlet temperature, and column flow.

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

1. Press [Front Det] or [Back Det] to open the FPD control table.
2. Set the detector temperature. The temperature must be greater than 120°C for the flame to light.
3. Change the hydrogen flow rate, if desired, and press [Off].
4. Change the air flow rate, if desired, and press [Off].
5. If you are using **packed columns**, turn off the makeup gas and proceed to Step 7.
6. If you are using **capillary columns**:
   a. Verify that makeup gas type is the same as that plumbed to your instrument (next to `Mkup` line of control table). Change the gas type, if necessary.
   b. If your capillary column is *defined*, choose a new flow mode, if desired, and set the makeup gas flow or combined flow.
   c. If your capillary column is *not defined*, enter a makeup gas flow. Only constant flow is available.
7. Scroll to `Flame` and press [On]. This turns on the air and hydrogen and initiates the ignition sequence. On ignition, the signal increases. Typical levels are 4 to 40 pA in sulfur mode, 10 to 70 pA in phosphorus mode. Verify that the flame is lit by holding a cold, shiny surface, such as a mirror or chrome-plated wrench, over the vent exit. Steady condensation indicates that the flame is lit.

Checkout Conditions and Chromatogram

This section contains typical examples of test sample chromatograms. It may be used as a general guide to instrument performance.

Note that injection volumes listed with operating conditions do not necessarily indicate total absolute volume injected. Volume given is simply the graduation (plunger position) read from a standard 10 µL syringe. For a heated inlet, actual sample volume injected will also include an additional 0.4 to 0.7 µL, the volume of sample volatilized from inside the syringe needle. For the dedicated, on-column inlet (unheated), the syringe plunger position more accurately reflects the true injected volume.

Also note that the following procedure and results are intended only to provide evidence of a properly functioning inlet and/or detector system; they are not necessarily suitable to test a given system against its specification limits.

FPD checkout conditions

Column and sample

Type	HP-5 30m × 0.32mm × 0.25 µm PN 19091J-413
Sample	FPD Checkout 8500-3697
Injection volume	1 µL

Inlet

Temperature	250° C Purged/Packed or Split/Splitless
	Oven Track Cool On-Column
	80°C PTV (see below)
Inlet pressure	25 psi (Constant pressure for EPC inlets, helium)

Split/Splitless

Mode	Splitless
Purge flow	60 mL/min
Purge time	0.75 min

Inlet, continued

PTV

Mode	Splitless
Inlet temperature	80°C
Initial time	0.1 min
Rate 1	720°C/min
Final temp 1	350°C
Final time 1	2 min
Rate 2	100°C/min
Final temp 2	250°C
Final time 2	0 min
Inlet pressure	25 psi (constant pressure for EPV inlets)
Purge time	0.75 min
Purge flow	60 mL/min

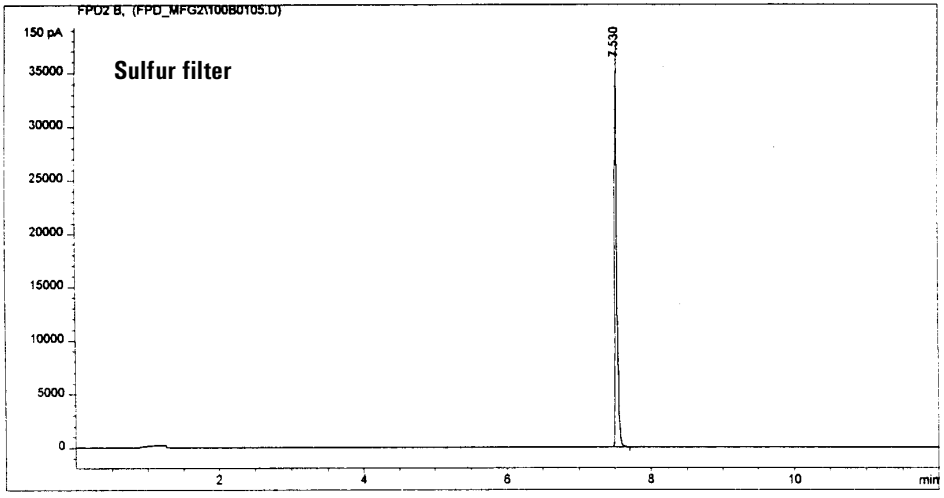
Detector

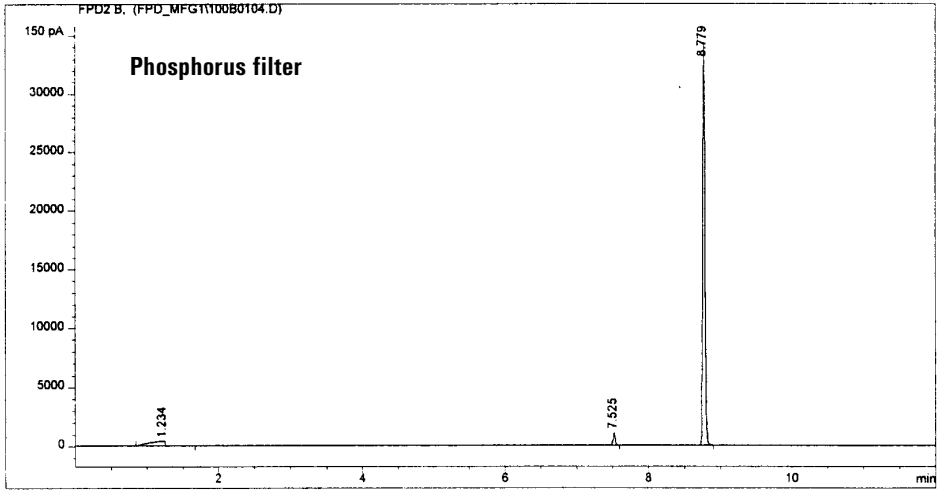
Temperature	200°C
Hydrogen flow	75 ± 2 mL/min
Air flow	100 ± 2 mL/min
Makeup flow	60 ± 2 mL/min, nitrogen
Offset, flow off (0-fa)	Should be < 40 display units
Offset, flame on (0 + fb)	< [(0-fa) + 85 display units]

Oven

Initial temp	60°C
Initial time	0 min
Rate 1	25°C/min
Final temp	110°C
Final time 0 min	
Rate 2	10°C/min
Final temp 2	170°C
Final temp 1	3 min

Typical FPD checkout chromatograms





Your retention times will differ, but peaks should resemble this example.

Maintaining the Detector

Caution Do not store the FPD at temperatures above 50°C, since this may damage the PMT.

Flame ignition problems

If the FPD flame won't light or stay lit, check/do the following:

1. Be sure there is a problem. Ignition is best confirmed by holding a mirror or shiny surface near the aluminum exhaust tube, with the rubber drip tube removed, and observing condensation if the flame is lit.
2. Check `Lit offset`. If it is zero, autoignition is turned off. If it is too large, the GC will not know that the flame is lit and will shut the detector down.
3. Increase the air supply pressure to the pneumatics module. This makes the flame easier to light but does not affect the air flow rate setpoint.
4. If the flame doesn't light at all, check the glow plug circuit. Observe the visual display, which will momentarily go to greater than 65500 counts when the flame lights. If the display doesn't change, check the pin connections at the printed circuit board, the lead connection on the glow plug and the appropriate 5A fuse on the GC main circuit board. If the glow plug has failed, replace it with part no. 0854-0141.
5. The flame is easier to light at higher detector temperatures.
6. Under some operating conditions, the flame may be more easily lit with the rubber drip tube removed. After lighting the flame, reinstall the drip tube.
7. If the flame still won't light, there could be a large leak in the system. This results in measured flow rates being different from actual flow rates, causing non-ideal ignition conditions. Thoroughly leak check the whole system.
8. If the analysis permits, substitute nitrogen for helium as carrier and makeup.
9. Increase hydrogen and air flow rates until ignition occurs, then reduce them toward the Table 69 values. Experiment for the best values.

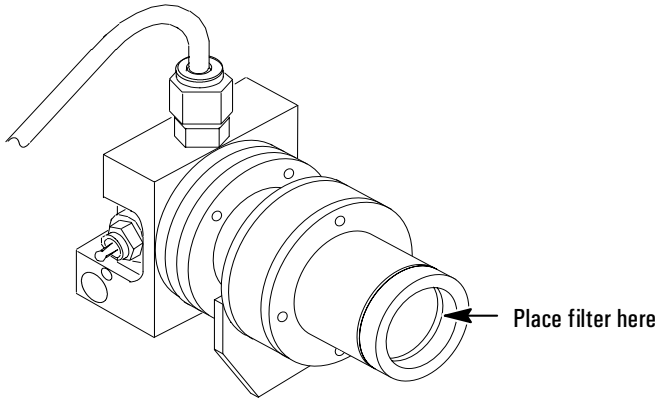
Changing wavelength filters

Install the correct optical filter, depending on the choice of Sulfur or Phosphorus mode. For Sulfur Mode, use the 393 nanometer filter (part no. 19256-80000). For Phosphorus Mode, use the 525 nanometer filter (part no. 19256-80010).

To change the filter:

WARNING Turn the main power switch—located under the left side of the oven door—off. If the photomultiplier tube is exposed to room light with the power on, it will be destroyed.

1. Release the retaining spring around the photomultiplier housing.
2. Pull the photomultiplier housing off the detector body. A twisting motion helps.
3. Remove the old filter. Use tissue to avoid fingerprints.
4. Place the new filter in the recess so that the silvered side faces the flame.



5. Push the PMT housing as far onto the detector body as it will go.
6. Install the retaining spring around the housing.
7. Restore power.

Leak testing

Turn off all supply gases. Cap the detector exhaust tube with a 1/4-inch Swagelok plug (part no. 0100-0196) and a 40% graphitized Vespel ferrule (part no. 0100-1061).

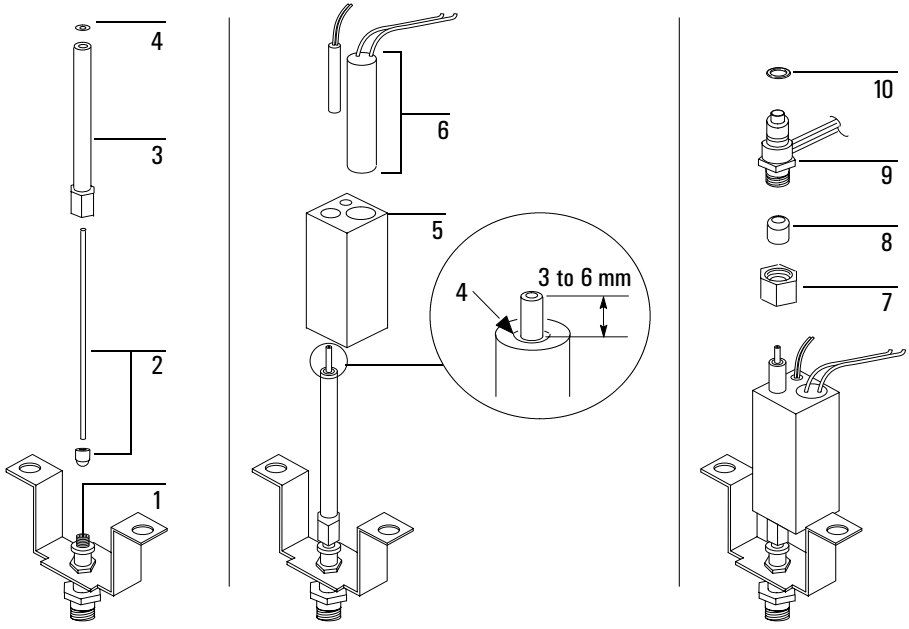
Caution When testing the flow system under pressure, do not exceed 210 kPa (30 psig). Higher pressures may damage the detector block window or seals.

Turn one of the gases on for a few seconds and then turn it off. The indicated flow-which is really a pressure-should remain constant or drop slowly. If not, there is a leak in the system. Begin checking possible leak sources and monitor the flow number to determine when the leak has been eliminated.

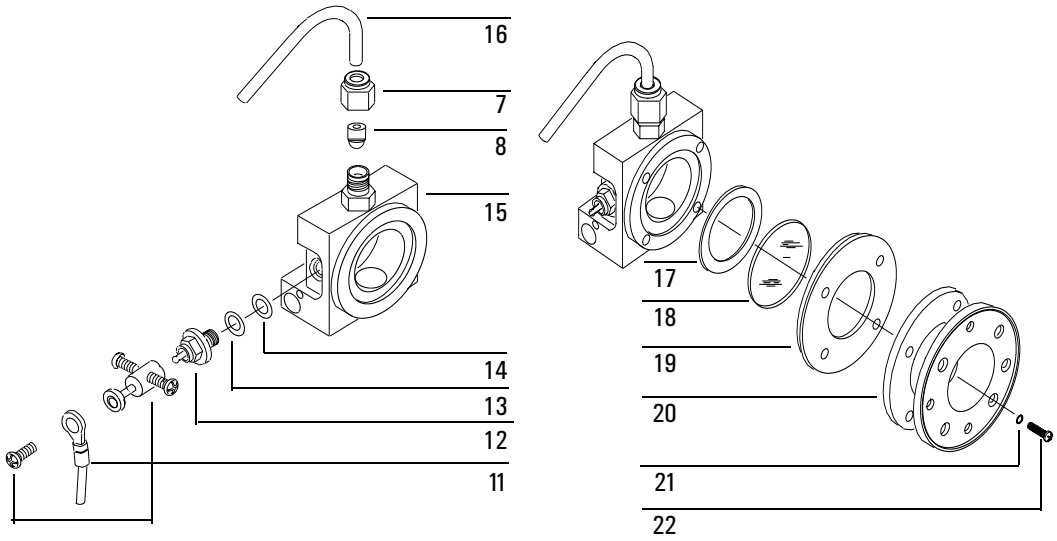
Possible leak sources, in order of decreasing probability, are:

1. Septum
2. Column fittings
3. Supply line swage-type plumbing connections
4. Detector block O-ring or Vespel seals
5. Other system plumbing

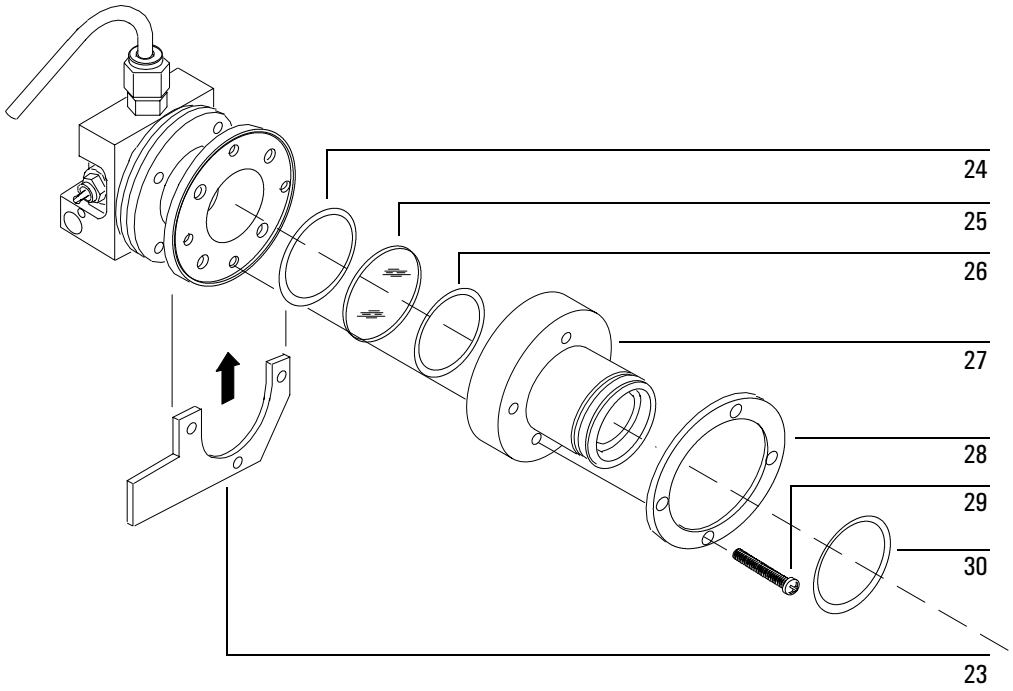
Parts identification



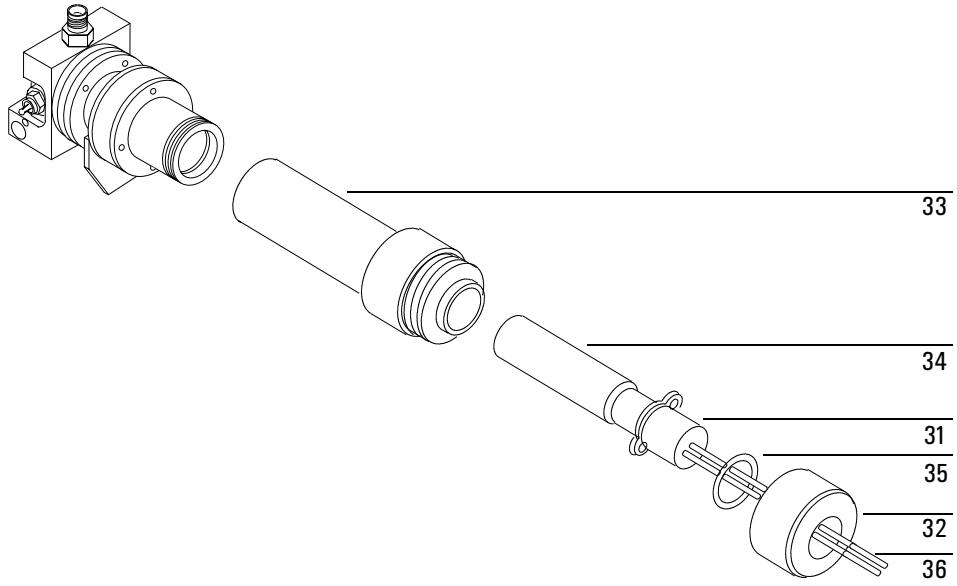
Item	Description	Part no.
1	Base assembly weldment	
2	Gigabore liner/ferrule assembly	19256-60590
3	Transfer tube	19256-80550
4	O-ring, Kalrez, transfer tube	0905-1101
5	Lower heater block	
6	Heater/sensor assembly	
7	Nut, brass, 1/4-inch	0100-0056
8	Ferrule, Vespel, 1/4-inch ID	5080-8774
9	Jet cartridge	G1535-80500
10	O-ring, Kalrez, jet cartridge	0905-1103



Item	Description	Part no.
7	Nut, brass, 1/4-inch	0100-0056
8	Ferrule, Vespel, 1/4-inch ID	5080-8774
11	Ignitor cable assembly	G1535-60600
12	Glow plug	0854-0141
13	Spacer, ignitor	19256-20590
14	O-ring, Kalrez, ignitor	0905-1102
15	Weldment, block	
16	Exit tube assembly, aluminum Exit tube assembly, stainless steel	19256-20700 19256-20705
17	Gasket, head shield	19256-80040
18	Window, first heat shield	19256-80030
19	Disk, heat shield	19256-20580
20	Coupling, stainless steel	19256-20550
21	Lockwasher (4 required)	2190-0108
22	Screw, M3 x 12 (4 required)	0515-0911



Item	Description	Part no.
23	Clamp	19256-00090
24	O-ring, silicone, 0.926-inch ID (orange)	0905-0955
25	Window, second heat shield	19256-80060
26	O-ring, silicone, 1.05-inch ID (orange)	0905-1104
27	Flange adapter	
28	Flange ring	19256-00200
29	Screw, M3 x 25 (4 required)	0515-0065
30	O-ring, Viton, 1.239-inch ID (brown)	0905-1100
Filters (not shown)		
	Sulfur mode	19256-80000
	Phosphorus mode	19256-80010



Item	Description	Part no.
31	Tube socket	19256-20670
32	End cap	19256-20710
33	PMT tube housing	19256-20650
34	Replacement photomultiplier tubes	
	PM tube ONLY	G1535-80050
	PM tube and housing assembly	19256-60510
35	O-ring for PM tube	0905-1099
36	Resistor network cable assembly	19256-60580

Cleaning/replacing windows, filters, and seals

Column bleed and/or effluent can contaminate the first quartz window (heat shield) nearest the detector module. Dust, fingerprints, atmospheric contaminants can dirty both quartz windows, the filter, and/or the photomultiplier tube window. Contamination anywhere along the light path between flame and PMT can reduce detector sensitivity.

1. Turn the electrometer off.
2. Turn hydrogen, air, and auxiliary gas supplies to the detector off. Turn the heaters off. Wait for the detector to cool.
3. Release the retaining spring around the photomultiplier housing.

Caution Always turn the electrometer off before removing the PMT housing to avoid destroying the tube.

Caution Keep the open end of the PMT housing covered as much as possible to avoid light damage to the tube.

4. Pull the PMT housing off the detector and remove the filter. Use lint-free lens tissue to clean the filter on both sides. Clean the PMT window seen inside the housing. Avoid scratching surfaces; do not use a cleaning fluid that might leave a film upon drying.
5. Inspect the filter: chips, scratches, and/or cracks in the light path scatter light, reducing detector sensitivity. Replace filters as necessary.

   Inspect the PMT window for damage; if necessary, replace the PMT.

   a. Remove the four screws in the PMT adapter flange and remove the flange. Use care as a quartz window is exposed and may fall out. Clean the window using lens tissue.

   b. Remove the four screws in the stainless steel coupling and carefully remove the coupling. The remaining quartz window may fall out. Clean the window using lens tissue.

Caution This window—the one closest to the flame—may stick when the detector is cold. It is easier to remove when the detector is warm, but be careful to avoid burns.

6. Note the placement and types of seals found on the disassembled parts. Seals should be replaced with new parts on reassembly.
7. Inspect the windows: chips, scratches, cracks or fogging in the light path scatter light, reducing sensitivity. Replace windows if necessary.
8. Reassemble the parts in reverse order, making sure all seals are of the proper type and in their proper locations. Tighten screws evenly and firmly to ensure gas- and light-tight seals. If the filter has a silvered side, it should face the flame (indicator arrows > on edge of filter should point toward the PMT).

Cleaning/replacing the jet

If a response problem is encountered (sensitivity, noise, selectivity) the FPD jet should be inspected for deposits and, if necessary, cleaned or replaced. To properly service the jet, the detector module should be removed from the instrument, followed by appropriate service:

1. Turn off power to the gas chromatograph and disconnect the main power cord. Remove the detector covers.
2. Allow time for the heated zones to cool to safe temperatures.

Caution Always turn the electrometer or the main power off before removing the PMT housing to avoid destroying the tube.

Caution Keep the open end of the PMT housing covered as much as possible to avoid light damage to the tube.

3. Remove the photomultiplier tube assembly from the detector module; also remove the filter. Set both in a safe place.
4. Remove the exhaust tubing.
5. Remove the sheet metal cover. On the single wavelength detector, it is held by two screws at the top and two at the bottom; on the dual wavelength detector it is held by two screws at the top. Loosen the screws holding the detector to the U-clamp. Use two wrenches to loosen the swage connection at the bottom of the jet assembly from the transfer line tube and carefully lift the burner module from the transfer tube so as not to damage the fused silica liner.

   It is not necessary to disconnect any plumbing, ignitor leads or the heater/sensor. Leave all attached and disconnect the detector block from the transfer line at the 1/4-inch swage fitting, then gently lift the block and rotate it enough to access the jet.

6. Remove and inspect the jet assembly. Rotating it slightly helps to free it. The jet assembly slips out of the FPD block more easily if the block is still warm. Use a wire or brush to remove any deposits.
7. This is also an ideal time to inspect/clean the glow plug (see “Flame ignition problems” on page 642), and inspect/clean the quartz windows (see ”Cleaning/replacing windows, filters, and seals”).
8. Use compressed air or nitrogen to blow out loose particles from the jet and/or detector module body.
9. Inspect and clean deposits from the jet bore using a suitable wire. If the jet is damaged in any way, replace it. It is good practice to replace the jet, rather than try to clean it, particularly when extremely high sensitivity is required.
10. Install a new Kalrez O-ring seal onto the jet. Do not re-use the old O-ring.

Caution Be careful not to crush or side-load the fused silica liner when reinstalling the detector.

11. Reassemble all parts of the detector module; reassemble the module onto the instrument. Use a new Vespel ferrule to seal the detector module to the transfer line.
12. Reinstall the PMT assembly on the detector module; restore instrument gases and power.

Replacing the transfer line fused silica liner

Occasionally the transfer line fused silica liner between the column and FPD module must be inspected, cleaned, and/or replaced.

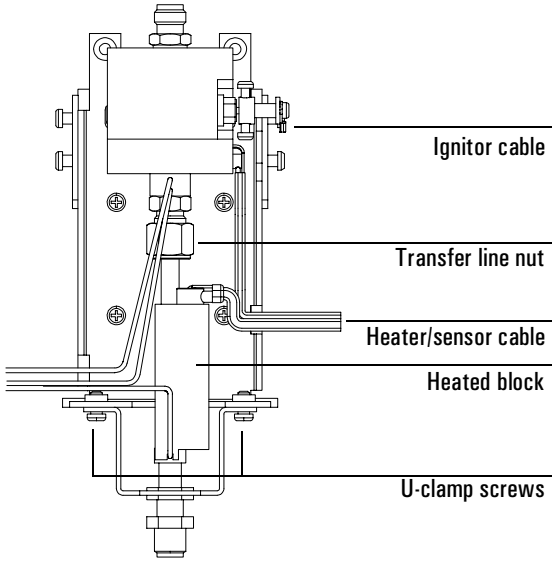
1. Turn off power to the gas chromatograph and disconnect the main power cord. Remove the detector covers.
2. Allow time for heated zones to cool to safe temperatures.
3. Inside the oven, remove the column to the FPD.

Caution Always turn the electrometer or the main power off before removing the PMT housing to avoid destroying the tube.

Caution In the next step, keep the open end of the PMT housing covered as much as possible to avoid light damage to the tube.

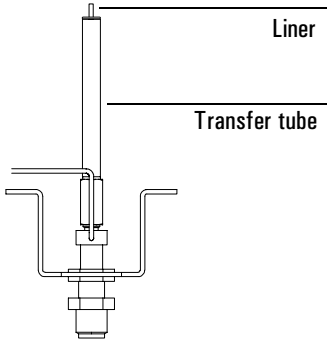
4. Remove the photomultiplier tube assembly—or assemblies—from the detector module; also remove the filter(s). Set them in a safe place.

5. Locate the ignitor cable attached to the side of the detector. Trace the cable back to the printed circuit board and disconnect it there.



6. Remove the exhaust tubing and the sheet metal cover—on the single wavelength detector, it is held by two screws on the top and two at the bottom; on the dual wavelength detector, it is held by two screws at the top.
7. Remove the four screws that attach the detector to the top of the oven (one at each corner). Remove the detector from the GC.
8. Loosen the transfer line nut. Remove the two screws that secure the U-clamp to the detector frame. Remove the U-clamp and the attached parts from the bottom of the detector.

9. Remove the transfer line nut and its ferrule, the heater/sensor cable assembly, and the heated block.



10. With an open end wrench, unscrew the transfer tube from the detector base. Lift the transfer tube—containing the fused silica liner—vertically off the instrument. Remove the fused silica liner and the 1/16-inch Vespel ferrule by pulling the liner and ferrule out from the bottom. Inspect for damage.
11. If necessary, install a new fused silica liner and Vespel ferrule. When doing so, carefully feed the fused silica liner through the Kalrez O-ring at the top of the transfer line so as not to damage the O-ring.
12. Carefully replace the fused silica liner, ferrule and tube onto the detector base. The fused silica liner should be positioned so that it protrudes 6 to 7 mm (1/4-inch) above the top of the transfer tube weldment. With a wrench, firmly tighten the transfer tube (1/2-turn past finger tight).
13. Reinstall the heated block, the heater/sensor cable assembly, the nut, and the ferrule. The notch in the bottom of the block fits over the tubing coming from the detector fitting.
14. Tighten the U-clamp screws, then tighten the nut on the transfer tube.
15. Place the detector on top of the instrument, orient it properly, and install the four screws to hold it. Install the top cover and the exhaust tubing.
16. Connect the ignitor cable to the printed circuit board.
17. Install the PMT assembly (or assemblies).
18. Restore normal operating conditions.

Replacing the photomultiplier tube

If the PMT is defective (high voltage on and the flame lit: low or no signal and/or high noise not attributed to any other source such as bad cables, light leaks, high temperature, defective signal board, etc.), it must be replaced.

1. Turn off power to the gas chromatograph and disconnect the main power cord.

Caution Turn the electrometer or main power off before opening the PMT housing to avoid destroying the tube.

2. Free the cables to the PMT from the clip on the support. Pull a few inches of the cables through the cable tie toward the end cap. Unscrew the end cap from the PMT assembly. Slide the cap away from the assembly.
3. Slide the resistor network cable assembly and the photomultiplier tube and socket out of the housing until about 1 inch of the tube is exposed.

Caution Protect the new PMT from light as much as possible to avoid damage to the tube.

4. Pull the socket off the PMT. Remove the PMT and replace with a new tube.
5. When seating the socket on the new tube, be certain that the missing pin on the tube base is lined up with the gap in the socket contacts.
6. Reassemble in reverse order. Make sure grease, fingerprints, dust, etc. are removed from the PMT window facing the detector module. Be sure that the O-ring is in place on the PMT/resistor bridge network assembly, as this is a critical light seal. If the O-ring is damaged, replace it.
7. Screw the end cap onto the PMT assembly. Pull the cables through the cable tie to eliminate slack at the end of the assembly. Place the cables in the clip on the side of the PMT housing support.

27 Basic Operations

Samples

Preparing the GC to run samples
Running samples - Manual Injection
Running samples - GC ALS or Valve Injection

Methods

Creating Methods
Setting a Column Flow Rate or Pressure

Sequences

Creating sequences
Procedure: Modifying a previously stored sequence
Start/Stop/Pause a sequence

Maintenance

Changing the Column
Checking Performance
Shutting down the GC

Samples

Preparing the GC to run samples

1. Check gas supplies and source pressures.
2. Check the power supply. Restore power if interrupted.
3. Turn on the GC, computer, and communication systems.
4. Check the identity of the installed column(s).
5. If needed, change the column. See "Changing the Column".
6. Check the availability of the samples to be analyzed.
7. Confirm what sequences and methods are required.

Running samples - Manual Injection

1. **Prepare the GC.** See "Preparing the GC to run samples".
2. **Prepare sample(s)** for injection.
3. **Load the desired method.** Press [Load] [Method], then input the desired method number and press [Enter]. See "Procedure: Loading a previously stored method".
4. **Wait for the Ready** prompt.
5. **Load the syringe**.
6. **Simultaneously** inject the sample and press [Start].

   The run light will come on and stay on until the run is completed.

Running samples - GC ALS or Valve Injection

1. **Prepare the GC**. See "Preparing the GC to run samples".
2. **Prepare sample(s)** for injection.
3. **Load sample vials** into the ALS tray or turret, if GC ALS is used. Remember the turret or tray position of each sample vial.
   - To edit injector setpoints, see "Procedure: Editing injector setpoints".
   - To configure the injector, see "Configuring the injector".
   - To edit the sample tray setpoints, see "Procedure: Editing the sample tray setpoints".
   - To configure the bar code reader, see "Procedure: Configuring the bar code reader".
4. **Load the desired sequence**. Press [Load] [Sequence]. Input the sequence number and press [Enter].
   - To create a sequence, see "Creating sequences".
   - To create a sampler subsequence, see "Procedure: Creating a sampler subsequence".
   - To create a valve subsequence, see "Procedure: Creating a valve subsequence".
   - To modify a sequence, see "Procedure: Modifying a previously stored sequence".
5. **Start the sequence**. Press [Seq control]. Scroll to `Start sequence`. Press [Enter].

   The Run light will come on and stay on until the sequence is completed.

Methods

Creating Methods

For more information, see "Analytical Methods".

1. **Set the oven parameters**. Press [Oven], and scroll down.
   - To create an isothermal run, see "Procedure: Setting up an isothermal run".
   - To create a single-ramp program, see"Procedure: Setting up a single-ramp program".
   - To create an oven program with up to six ramps, see "Procedure: Setting up a multiple-ramp program".
2. **Set your column parameters**. Press [Col 1] or [Col 2] and enter:
   a. The column length and diameter (capillary columns). See "Procedure: Configuring a capillary column".
   b. A column mode, if available. See "Procedure: Selecting a column mode".
   c. The column head pressure or column flow. See "Procedure: Configuring a capillary column", "Procedure: Selecting a column mode", or "Procedure: Setting initial flow or pressure or average linear velocity".
3. **Set the inlet parameters**. Press [Front Inlet] or [Back Inlet].
   - Select inlet mode, if available.
   - Enter parameters. For example, set the temperature, pressure, split ratio, split flow, and total flow. See "Using a Split/Splitless Inlet", "Using a Purged Packed Inlet", "Using a Cool On-Column Inlet", "Introducing the Agilent PTV", or "Using a Volatiles Interface".
4. **Set the detector parameters**. Press [Front Det] or [Back Det].
   - Enter parameters. For example, set temperature, hydrogen flow, air flow, makeup gas and flow. See "Operating with EPC", "Operating the TCD",

"Operating with EPC", "Procedure: Operating the μ-ECD", or "Operating the FPD".

5. **Save these parameters as a method**. Press [Store] [Method]. Input a method number (1 through 9), then press [Enter].

Setting a Column Flow Rate or Pressure

1. Set the new value in the column control table. See "Procedure: Setting initial flow or pressure or average linear velocity".
2. To save the change, press [Store] [Method]. Input a method number (1 through 9), and press [Enter].

Sequences

Creating sequences

To create a sequence

1. **Press [Seq]** to open the sequence control table.

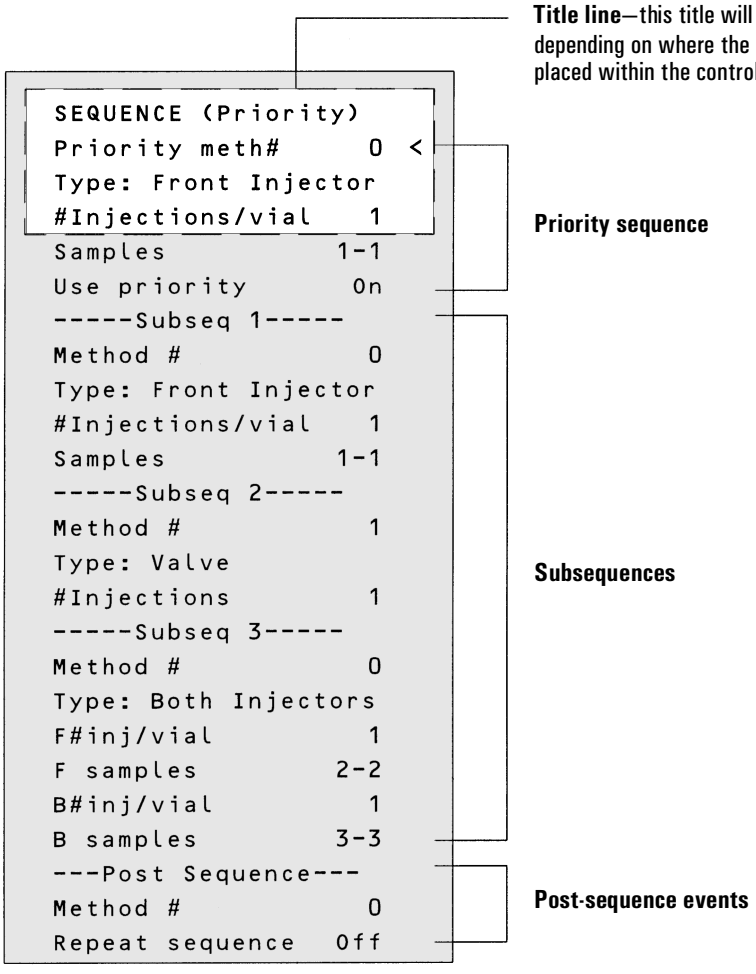


Figure 92 Example sequence table

2. **Scroll to `SEQUENCE (Priority)`**. Use the priority sequence (set it to `On`) only if you want to interrupt a running sequence to run urgent samples now. Otherwise set it to `Off`. See "Priority sequence".
3. **Scroll to `Subseq 1`**. Set the sequence type (sampler or valve) using the [Mode/Type] key. Define the samples to be run, where they are located, and what method to use for them.
   - For details about creating a sampler subsequence, see "Procedure: Creating a sampler subsequence".
   - For details about creating a valve subsequence, see "Procedure: Creating a valve subsequence".
   - For general information about sequences, see "Analytical Sequences".
4. **Create additional subsequences** as needed.
   - Create a subsequence for each set of valve or automatic liquid sampler samples you wish to run with a given method.
   - Create a subsequence for each set of samples that require a different method.
   - You can create up to five subsequences.
5. **Set any desired post sequence events**. Scroll to `SEQUENCE (Post Seq)`.
   - Input the Post Sequence method number. Enter `0` to not load a method.
   - To repeat the sequence indefinitely, turn `Repeat Sequence` On.
   - See "Post Sequence" or "Procedure: Setting the Post Sequence events" for more information.
6. **Store the completed sequence**. See "Procedure: Storing a sequence".

For more information, see "Analytical Sequences".

Start/Stop/Pause a sequence

To control a sequence, access the Sequence control table by pressing [Seq Control].

What do you want to do?	See
Start a sequence	Procedure: Starting/running a sequence
Stop a sequence after the current run	Procedure: Stopping a sequence
Pause a sequence	Procedure: Pausing and resuming a sequence
Run a priority sample now, then continue the sequence	Priority sequence
Abort the run and sequence immediately	Press the [Stop] key to immediately halt the current run and the sequence. See Aborting a sequence for more details.

Maintenance

Changing the Column

To change a column:

1. **Select the appropriate fittings** and adapters for your:
   - Capillary columns, see "Ferrules for capillary columns".
   - Packed metal columns, see "Fittings" and "Ferrules for packed metal columns".
   - Packed glass columns, see "Ferrules and O-rings for glass packed columns".
2. **Prepare your column**.
   - If using a capillary column, see "Procedure: Preparing capillary columns".
   - If using a packed metal column, see "Preparing packed metal columns".
3. **Lower the temperatures** of the oven, inlet, and detector to <40°C.
4. **Check the liner**. Be sure you have the correct liner (or other inlet hardware) installed. For instructions on choosing and installing liners, see "Liners".

Inlet	Refer To
Split/splitless	"Liners"
Purged packed	"Liners and inserts"
Cool on-column	"Hardware"
Programmed temperature vaporization	"Inlet adapters"

5. **Install the column**.

Column Type	Inlet or Detector	Refer To
Capillary	Split/splitless inlet	"Procedure: Installing capillary columns in the split/splitless inlet"
	Cool on-column inlet	"Procedure: Installing capillary columns in the cool on-column inlet"
	Purged packed inlet	"Procedure: Installing capillary columns in the purged packed inlet"
	Programmed temperature vaporization inlet and Volatiles interface	"Procedure: Installing capillary columns in the PTV inlet and Volatiles Interface"
	FID NPD	"Procedure: Installing capillary columns in NPD and FID detectors"
	TCD	"Procedure: Installing capillary columns in the TCD"
	µ-ECD	"Procedure: Installing capillary columns in the µ-ECD"
	FPD	"Procedure: Installing capillary columns in the FPD"
Packed Metal	Any	"Procedure: Installing an adapter in a detector fitting" and "Procedure: Installing packed metal columns"
Packed Glass	Any	"Procedure: Installing glass packed columns"

6. **Condition your column**, if needed. See "Conditioning columns".
7. If using a capillary column, configure (define) it if desired. See "Procedure: Configuring a capillary column".

Now may be a good time to check your inlet septum and change it if needed.

Checking Performance

To check the performance of your GC, run the recommended sample mixture for your detector type as described below.

1. **Install the checkout column**. For the FID, TCD, NPD, µ-ECD, and FPD, use an HP-5, 30 m x 0.32 mm x 0.25 µm capillary column (part number 19091J-413).

2. **Install the appropriate liner or insert**, if needed:

Inlet	Item
Split/splitless	Liner, part no. 5062-3587 (splitless)
Purged packed	Liner, part no. 5181-3382 (deactivated)
Cool on-column	Insert, part no.19245-20525
Programmed temperature vaporization	Baffled liner, part no. 5183-2037 320 μm adapter part no. 5182-9761

3. **Set the checkout conditions** on your GC.

Detector Type:	Refer to:
FID	"FID checkout conditions"
TCD	"TCD checkout conditions"
NPD	"NPD checkout conditions"
μ-ECD	"μ-ECD checkout conditions"
FPD	"FPD checkout conditions"

4. **Prepare your sample(s)**.

5. When the GC is ready, **make the injection and start the run**.

   - If using manual injection, see "Running samples - Manual Injection".
   - If using sampler injection, see "Running samples - GC ALS or Valve Injection".

6. **Compare your result** against the appropriate reference chromatogram:

Detector Type:	Refer to:
FID	"Typical FID checkout chromatogram"
TCD	"Typical TCD checkout chromatogram"
NPD	"Typical NPD checkout chromatogram"
μ-ECD	"Typical μ-ECD checkout chromatogram"
FPD	"Typical FPD checkout chromatograms"

Remember that the reference chromatogram is typical and is only intended to serve as a guide.

28 Site Preparation

Temperature and humidity ranges

Ventilation requirements
- Venting oven exhaust
- Venting toxic or noxious gases

Benchtop space requirements

Electrical requirements
- Grounding
- Line voltage
- USA fast heating oven, 240 V
- Canadian installation
- Configuring the GC for an MSD

Gas requirements
- Gases for packed columns
- Gases for capillary columns
- Gas purity

The gas plumbing
- Supply tubing for carrier and detector gases
- Two-stage pressure regulators
- Pressure regulator-gas supply tubing connections
- Traps

Cryogenic cooling requirements
- Choosing a coolant
- Using carbon dioxide
- Using liquid nitrogen

Supplying valve actuator air

Site preparation at a glance

Before the GC arrives, make sure your laboratory meets the following environmental, weight, power, and gas requirements. You should also refer to this checklist for supplies that you need to operate your GC, such as traps and tubing. You can find more site preparation information in this chapter.

Site Preparation Checklist

- ❒ The site is well ventilated and free of corrosive materials and overhanging obstacles.
- ❒ Site temperature is within the recommended range of 20 to 27° C.
- ❒ Site humidity is within the recommended range of 50 to 60%.
- ❒ Bench space is adequate for the GC with EPC: 50 cm x 58.5 cm x 50 cm (21 inch x 23 inch x 21 inch).
  Bench space is adequate for the GC without EPC: 50 cm x 68 cm x 50 cm (21 inch x 26.7 inch x 21 inch).
- ❒ Bench can support the weight of the 6890 system. See page 675.
- ❒ Power receptacle is earth grounded. See page 676.
- ❒ Electrical supply meets all GC's power requirements. See page 676.
- ❒ Voltage supply is adequate for oven type. **Regular oven:** 2,250 VA. **Fast-heating oven:** 2,950 VA.
- ❒ Gas supplies meet the requirements of your columns and detectors. See page 679.
- ❒ Gases meet purity requirements. All should be chromatographic-grade—99.9995% pure or better. Air should be zero-grade or better. Detector air is not shared with valve actuators.
- ❒ Precleaned, 1/8-inch (or 1/4-inch) copper tubing is available for connecting inlet and detector gas supplies. See page 685.
- ❒ Inlet and detector gas supplies have two-stage pressure regulators installed.

Optional supplies:

- ❒ High quality traps for inlet and detector gas supplies—molecular sieve trap, hydrocarbon trap, and/or oxygen trap.
- ❒ Liquid N_2 or liquid CO_2 (depending on requirements) available for cryogenic cooling.
- ❒ Supply of 1/4-inch, insulated copper tubing is available for liquid N_2 supplies, **OR**
  1/8-inch, heavy-walled, stainless steel tubing is available for liquid CO_2 supplies.
- ❒ Insulation for liquid N_2 tubing is available.
- ❒ Pressurized clean air is available for value actuators. See page 692.

Site Preparation

Site preparation involves two general steps: insuring that your laboratory is capable of supporting the operation of the GC and obtaining supplies and tools you will need to install the instrument. A list of the necessary tools and supplies appears at the beginning of "Installation". Most supplies are available from Agilent Technologies. See the Agilent catalog for consumables and supplies for descriptions and ordering information. You can obtain a copy of the catalog from your local sales office.

Temperature and humidity ranges

Operating the GC within the recommended ranges insures optimum instrument performance and lifetime.

Recommended temperature range	Temperature range
20 to 27° C	5 to 40° C
Recommended humidity range	**Humidity range**
50 to 60%	Up to 31° C, 5 to 80% At 40° C, 5 to 50%
Recommended altitude range	
Up to 2000 m	

After exposing the GC to extremes of temperature or humidity, allow 15 minutes for it to return to the recommended ranges.

Ventilation requirements

The GC is cooled by convection: air enters vents in the side panels and underneath the instrument. Warmed air exits through slots in the top, rear, and side panels. Do not obstruct air flow around the instrument.

Caution For proper cooling and general safety, always operate the instrument with cover panels properly installed.

Venting oven exhaust

Hot air (up to 450°C) from the oven exits through a vent in the rear. Allow at least 20 cm (10 inch) clearance behind the instrument to dissipate this air.

WARNING Do not place temperature-sensitive items (for example, gas cylinders, chemicals, regulators, and plastic tubing) in the path of the heated exhaust. These items will be damaged and plastic tubing will melt. Be careful when working behind the instrument during cool-down cycles to avoid burns from the hot exhaust.

If space is limited, the Oven Exhaust Deflector (part no. 19247-60510) may improve oven cooling. It diverts exhaust air up and away from the instrument. You can connect it to a 10.2-cm (4-inch) exhaust-duct system, route the exhaust to a fume hood, or vent the exhaust outside the building with 10.2-cm diameter (4-inch diameter) furnace duct.

Venting toxic or noxious gases

During normal operation of the GC with many detectors and inlets, some of the carrier gas and sample vents outside the instrument. If any sample components are toxic or noxious, or if hydrogen is used as the carrier gas, the exhaust must be vented to a fume hood. Place the GC in the hood or attach a large diameter venting tube to the outlet for proper ventilation.

To further prevent contamination from noxious gases, you can attach a chemical trap (part no. G1544-60610) to the split vent.

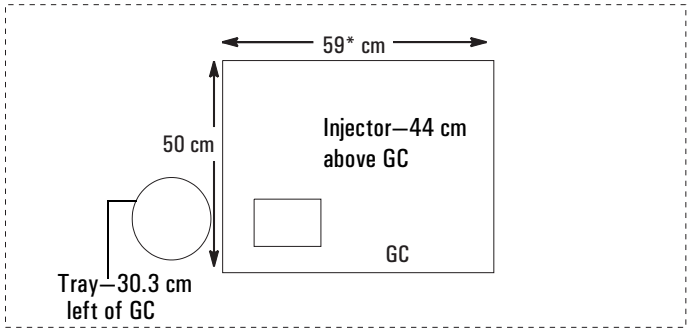
Benchtop space requirements

The GC with electronic pneumatics control (EPC) is 59 cm (23 inch) wide. The nonEPC model is 68 cm (26.7 inch) wide. Both are 50 cm (21 inch) high and 50 cm (21 inch) deep.

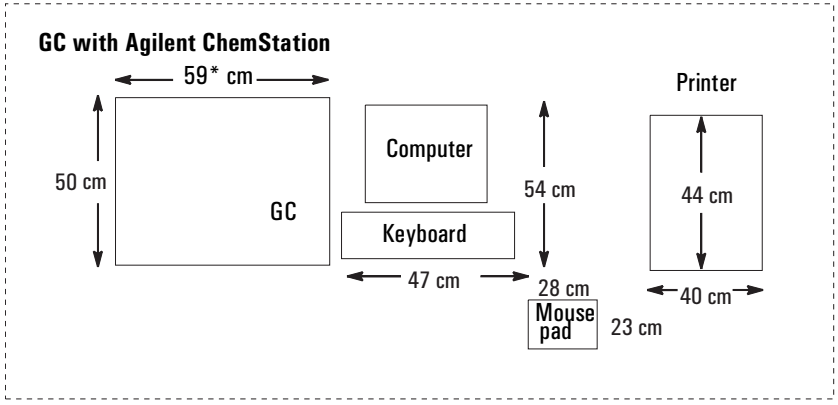
The area above the GC should be clear, with no shelves or overhanging obstructions that limit access to the top of the instrument and interfere with cooling. You may need additional space for other instruments used with your GC. Figure 93 shows some common system configurations.

Table 70 presents the dimensions, power requirements, heat production, and weight of the GC and other Agilent instruments often used with it. Use this table to insure that you have adequate space and power for the entire system. Allow at least 10.2 cm (4 inch) space between instruments for ventilation. See Table 71 and Table 72 for GC voltage requirements.

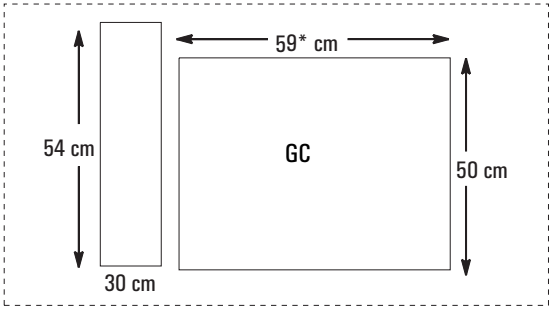
GC with Agilent Automatic Liquid Sampler



GC with Agilent ChemStation



GC with 5973N Mass Selective Detector



*68 cm for non-EPC version.

Figure 93. Common GC system configurations—top views

Table 70. Dimensions, Power, Heat Production, and Weight

Instrument	Height	Width	Depth	Power (VA)	Heat	Weight
6890 Gas Chromatograph						
EPC version	54 cm 21 inch	59 cm 23 inch	54 cm 21 inch	2,250	8,100 KJoules 7,681 Btu/hr	50 kg 112 lb
Non-EPC version	51 cm 21 inch	68 cm 26.7 inch	54 cm 21 inch	2,250	8,100 KJoules 7.681 Btu/hr	56.8 kg 125 lb
Fast heating oven same for EPC and non-EPC	—	—	—	2,950	10,620 KJoules 10,071 Btu/hr	—
GC Automatic Liquid Sampler						
G2613A Injector	44 cm above GC 17 inch above GC					
G2614A Tray	30.3 cm left of GC 9 inch left of GC					
Computer*						
Computer with monitor	54 cm 21 inch	42 cm 17 inch	39 cm 15 inch	N/A	N/A	N/A
Keyboard	5 cm 2 inch	47 cm 18 inch	18 cm 7 inch	N/A	N/A	N/A
5973N Mass Selective Detector	35 cm 13.6 inch	17 cm 6.7 inch	65 cm 25.6 inch	254 max	3,158 Btu/hr, 3,000 with GC	22.7 kg 50.0 lb
7694 Headspace Sampler	31 cm 16 inch	56 cm 22 inch	39 cm 22 inch	420 max	2,215 KJoules 2,100 Btu/hr	35.8 kg 79.0 lb
Printers						
Laser printer	41 cm 16 inch	30 cm 12 inch	54 cm 22 inch	300 max	N/A	39 kg 85 lb
Integrators						
3397 Series, 3396 Series III and, 3395 Integrators	13 cm 4.5 inch	46 cm 18 inch	46 cm 18 inch	50	135 KJoules 120 Btu/hr	4.3 kg 9.5 lb
35900C/D/E Analog-to-Digital Converter	11 cm 4 inch	33 cm 13 inch	29 cm 11 inch	40	216 KJoules 205 Btu/hr	4.1 kg 9.0 lb

* General specifications for a mid-size, desktop computer

Electrical requirements

Grounding

Caution A proper earth ground is required for GC operations.

To protect users, the metal instrument panels and cabinet are grounded through the three-conductor power line cord in accordance with International Electrotechnical Commission (IEC) requirements.

The three-conductor power line cord, when plugged into a properly grounded receptacle, grounds the instrument and minimizes shock hazard. A properly grounded receptacle is one that is connected to a suitable earth ground. Proper receptacle grounding should be verified.

Make sure the GC is connected to a dedicated receptacle. Use of a dedicator receptacle reduces interference.

Caution Any interruption of the grounding conductor or disconnection of the power cord could cause a shock that could result in personal injury.

Line voltage

The GC operates from one of the AC voltage supplies listed in Table 71, depending on the standard voltage of the country from which it was ordered. GCs are designed to work with a specific voltage; make sure your GC voltage option is appropriate for your lab. The voltage requirements for your GC are printed near the power cord attachment.

Table 71. Line Voltage Requirements

Voltage	Maximum power consumption (VA)	Power line requirement	Oven type
120 V (±5%)	2,250	20-amp dedicated	Slow-heating
200 V (±5%)	2,950	15-amp dedicated	Fast-heating
208 V (±5%)	2,950	15-amp dedicated	Fast-heating
220 V (±5%)	2,950	15-amp dedicated	Fast-heating
230 V (±5%)	2,950	16-amp dedicated	Fast-heating
230 V (±5%)	2,250	10-amp dedicated	Slow-heating
(Switzerland or Denmark with 10-amp maximum service)			
240 V (±5%)	2,950	13- or 16-amp dedicated	Fast-heating

Frequency range for all voltages is 48 to 66 Hz.

The fast-heating oven requires at least 200 V. Most countries' standard voltage meets this requirement. GCs for use in the USA, Denmark, and Switzerland will be equipped with a slow-heating oven unless they are ordered with power option 002, which specifies a fast-heating oven.

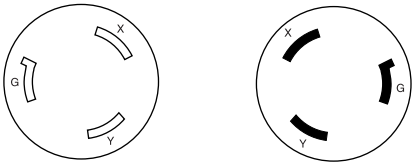
Although your GC should arrive ready for operation in your country, compare its voltage requirements with those listed in Table 3. If the voltage option you ordered is not suitable for your installation, contact Agilent Technologies.

Table 72. Voltage Requirements by Country

Country	Voltage	Oven type
Australia, 10 amp	240 V	Slow–heating
Australia, India, South Africa	240 V	Fast–heating
China	220 V	Slow–heating
China, Hong Kong	220 V	Fast–heating
Continental Europe, dual phase	230 V	Fast–heating
Continental Europe, single phase	220 V	Fast–heating
Denmark, Switzerland, 10 amp	230 V	Slow–heating
Denmark, Switzerland, 16 amp	230 V	Fast–heating
Israel	220 V	Fast–heating
Japan	200 V	Fast–heating
United Kingdom, Ireland	240 V	Fast–heating
USA	120 V	Slow–heating
USA	208 V	Fast–heating
USA	240 V	Fast–heating

USA fast heating oven, 240 V

The 240 V fast heating oven requires 240 V/15A power. Do not use 208 V power. Lower voltage causes slow oven ramps and prevents proper temperature control. The power cord supplied with your GC is rated for 250 V/15A, and is a two pole, three wire cord with grounding (type L6-15R/L6-15P). See the figure below.



Receptacle
L6-15R

Plug
L6-15P

Canadian installation

When installing a GC in Canada, make sure your GC's power supply circuit meets the following additional requirements:

- The circuit breaker for the branch circuit, which is dedicated to the instrument, must be rated for continuous operation.
- The service box branch circuit must be marked as a "Dedicated Circuit."

Configuring the GC for an MSD

If you are installing an Agilent Mass Selective Detector, you must configure the GC to properly control the heated transfer line.

1. Press [Config][Aux], and select [1] if the MSD is installed in the front position or [2] for the back position.
2. Press [Mode/Type].
3. Use the scroll keys to select `MSD` as the Aux zone type. Press [Enter].

If you do not configure the Aux zone for MSD, Warning 101, *Invalid heater power for front (back) detector, inlet, and aux 1(2)*, will appear on the GC display, and the heated zones will be set to *not installed*.

Gas requirements

Gases for packed columns

The carrier gas you use depends upon the type of detector and the performance requirements. Table 73 lists gas recommendations for packed column use. In general, makeup gases are not required with packed columns.

Table 73. Gas Recommendations for Packed Columns

Detector	Carrier gas	Comments	Detector, anode purge, or reference gas
Electron Capture	Nitrogen	Maximum sensitivity	Nitrogen
	Argon/Methane	Maximum dynamic range	Argon/Methane
Flame Ionization	Nitrogen	Maximum sensitivity	Hydrogen and air for detector
	Helium	Acceptable alternative	
Flame Photometric	Hydrogen		Hydrogen and air for detector
	Helium		
	Nitrogen		
	Argon		
Nitrogen-Phosphorus	Helium	Optimum performance	Hydrogen and air for detector
	Nitrogen	Acceptable alternative	
Thermal Conductivity	Helium	General use	Reference must be same as carrier
	Hydrogen	Maximum sensitivity (Note A)	
	Nitrogen	Hydrogen detection (Note B)	
	Argon	Maximum hydrogen sensitivity (Note B)	

Note A: Slightly greater sensitivity than helium. Incompatible with some compounds.
Note B: For analysis of hydrogen or helium. Greatly reduces sensitivity for other compounds.

Gases for capillary columns

When used with capillary columns, GC detectors require a separate makeup gas for optimum sensitivity. For each detector and carrier gas, there is a preferred choice for makeup gas. Table 74 lists gas recommendations for capillary columns.

Table 74. Gas Recommendations for Capillary Columns

Detector	Carrier gas	Preferred makeup gas	Second choice	Detector, anode purge, or reference gas
Electron Capture	Hydrogen	Argon/Methane	Nitrogen	Anode purge must be same as makeup
	Helium	Argon/Methane	Nitrogen	
	Nitrogen	Nitrogen	Argon/Methane	
	Argon/Methane	Argon/Methane	Nitrogen	
Flame Ionization	Hydrogen	Nitrogen	Helium	Hydrogen and air for detector
	Helium	Nitrogen	Helium	
	Nitrogen	Nitrogen	Helium	
Flame Photometric	Hydrogen	Nitrogen		Hydrogen and air for detector
	Helium	Nitrogen		
	Nitrogen	Nitrogen		
	Argon	Nitrogen		
Nitrogen-Phosphorus	Helium	Nitrogen	Helium**	Hydrogen and air for detector
	Nitrogen	Nitrogen	Helium**	
Thermal Conductivity	Hydrogen*	Must be same as carrier and reference gas	Must be same as carrier and reference gas	Reference must be same as carrier and makeup
	Helium			
	Nitrogen			

* When using hydrogen with a thermal conductivity detector, vent the detector exhaust to a fume hood or a dedicated exhaust to avoid buildup of hydrogen gas.

**Helium is not recommended as a makeup gas at flow rates > 5 mL/min. Flow rates above 5 mL/min shorten detector life.

Gas purity

Some gas suppliers furnish “instrument” or “chromatographic” purity grades of gas that are specifically intended for chromatographic use. We recommend these grades for use with the GC.

Generally, all gas supplies used should be in the 99.995% to 99.9995% purity range. Only very low levels (≤ 0.5 ppm) of oxygen and total hydrocarbons should be present. Oil-pumped air supplies are not recommended because they may contain large amounts of hydrocarbons.

The addition of high-quality moisture and hydrocarbon traps immediately after the main tank pressure regulator is highly recommended. Refer to the next section, "Assembling the Gas Plumbing," for more information on using traps.

Table 75. Gas Purity Recommendations

Carrier gases and capillary makeup gases	
Helium	99.9995%
Nitrogen	99.9995%
Hydrogen	99.9995%
Argon/Methane	99.9995%
Detector support gases	
Hydrogen	99.9995%
Air (dry)	Zero-grade or better

Table 76. Maximum/Minimum inlet and detector pressures

Detector or Inlet	Gas type	Maximum Pressure kPa	Maximum Pressure psi	Minimun Pressure kPa	Minimun Pressure psi
FID	H2	690	100	240	35
	Air	690	100	380	55
	Make up	690	100	380	55
NPD	H2	690	100	240	35
	Air	690	100	380	55
	Make up	690	100	380	55
TCD	Make up	690	100	380	55
	Reference	690	100	380	55
ECD	Make up	690	100	380	55
FPD	H2	690	100	310	45
	Air	827	120	690	100
	Make up	690	100	380	55
Split/splitless	150 psi	1172	170	All inlets: 38 kPa (20 psi) above pressure used in method	
	100 psi	827	120		
On-column		827	120		
Purged Packed		827	120		
PTV		827	120		

An addition 20 psi above desired column head pressure is required to maintain column flow.

The gas plumbing

WARNING All compressed gas cylinders should be securely fastened to an immovable structure or permanent wall. Compressed gases should be stored and handled in accordance with the relevant safety codes.

Gas cylinders should not be located in the path of heated oven exhaust.

To avoid possible eye injury, wear eye protection when using compressed gas.

Follow the general plumbing diagram in when preparing gas supply plumbing.

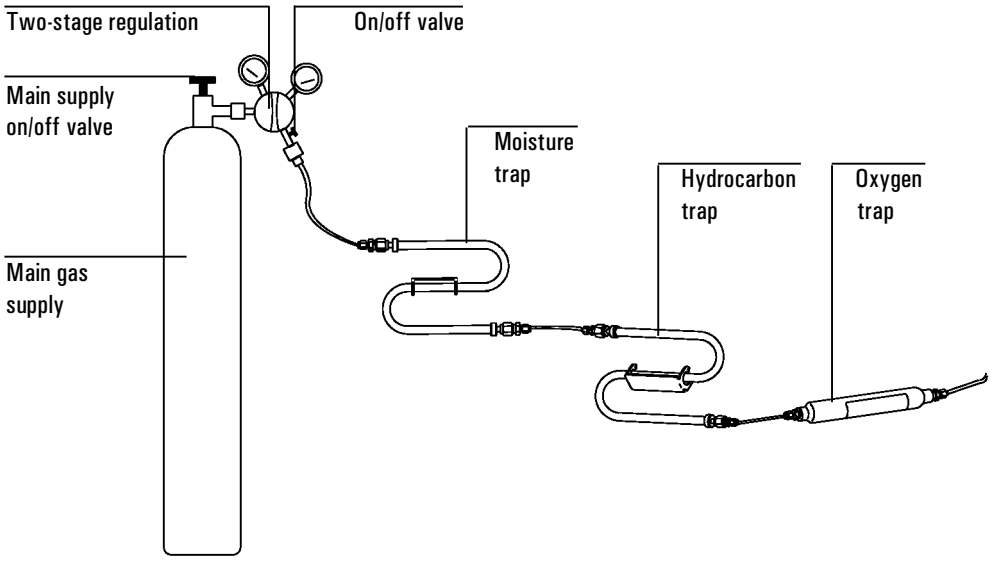


Figure 94. General plumbing diagram

- Two-stage regulators are strongly recommended to eliminate pressure surges. High-quality, stainless-steel diaphragm-type regulators are especially recommended.
- On/off valves mounted on the outlet fitting of the two-stage regulator are not essential but are very useful. Be sure the valves have stainless-steel, packless diaphragms.
- FID, FPD, and NPD detectors require a dedicated air supply. Operation may be affected by pressure pulses in air lines shared with other devices.
- Flow- and pressure-controlling devices require at least 10 psi (138 kPa) pressure differential across them to operate properly. Source pressures and capacities must be high enough to ensure this.
- Auxiliary pressure regulators should be located close to the GC inlet fittings. This insures that the supply pressure is measured at the instrument rather than at the source; pressure at the source may be different if the gas supply lines are long or narrow.

Supply tubing for carrier and detector gases

Caution Do not use methylene chloride or other halogenated solvent to clean tubing that will be used with an electron capture detector. They will cause elevated baselines and detector noise until they are completely flushed out of the system.

Gases should be supplied to the instrument only through preconditioned copper tubing (part no. 5180-4196). Do not use ordinary copper tubing—it contains oils and contaminants.

Caution Do not use plastic tubing for suppling detector and inlet gases to the GC. It is permeable to oxygen and other contaminants that can damage columns and detectors, and can melt if near hot exhaust or components.

The tubing diameter depends upon the distance between the supply gas and the GC and the total flow rate for the particular gas. One-eighth-inch tubing is adequate when the supply line is less than 15 feet (4.6 m) long.

Use larger diameter tubing (1/4-inch) for distances greater then 15 feet (4.6 m) or when multiple instruments are connected to the same source. You should also use larger diameter tubing if high demand is anticipated (for example, air for an FID).

Be generous when cutting tubing for local supply lines—a coil of flexible tubing between the supply and the instrument lets you move the GC without moving the gas supply. Take this extra length into account when choosing the tubing diameter.

Two-stage pressure regulators

To eliminate pressure surges, use a two-stage regulator with each gas tank. Stainless steel, diaphragm-type regulators are recommended.

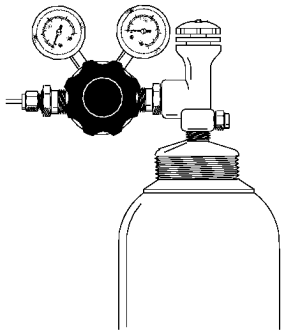


Figure 95. Two-stage pressure regulator

The type of regulator you use depends upon gas type and supplier. The Agilent catalog for consumables and supplies contains information to help you identify the correct regulator, as determined by the Compressed Gas Association (CGA). Agilent Technologies offers pressure-regulator kits that contain all the materials needed to install regulators properly.

Pressure regulator-gas supply tubing connections

The pipe-thread connection between the pressure regulator outlet and the fitting to which you connect the gas tubing must be sealed with Teflon tape.
Instrument grade Teflon tape (part no. 0460-1266), from which volatiles have

been removed, is recommended for all fittings. Do not use **pipe dope** to seal the threads; it contains volatile materials that will contaminate the tubing.

Traps

Using chromatographic-grade gases insures that the gas in your system is pure. However, for optimum sensitivity, it is highly recommended that you install high-quality traps to remove traces of water or other contaminants. After installing a trap, check the gas supply lines for leaks.

Table 77. Recommended Traps

Description	Part no.
Preconditioned moisture trap: metal casing, s-shaped trap for carrier gas cleanup. Contains Molecular Sieve 5A, 45/60 mesh, and 1/8-inch fittings.	5060-9084
Hydrocarbon trap: metal casing, s-shaped trap filled with 40/60 mesh activated charcoal, and 1/8-inch fittings	5060-9096
Oxygen trap (for carrier and ECD gases): metal casing, and 1/8-inch brass fittings. Oxygen trap cannot be reconditioned.	3150-0414

Moisture in carrier gas damages columns. We recommend a type 5A Molecular Sieve trap after the source regulator and before any other traps.

A hydrocarbon trap removes organics from gases. It should be placed after a molecular sieve trap and before an oxygen trap, if they are present.

An oxygen trap removes 99% of the oxygen from a gas plus traces of water. It should be last in a series of traps. Because trace amounts of oxygen can damage columns and degrade ECD performance, use an oxygen trap with carrier and ECD gases. Do not use it with FID, FPD, or NPD fuel gases.

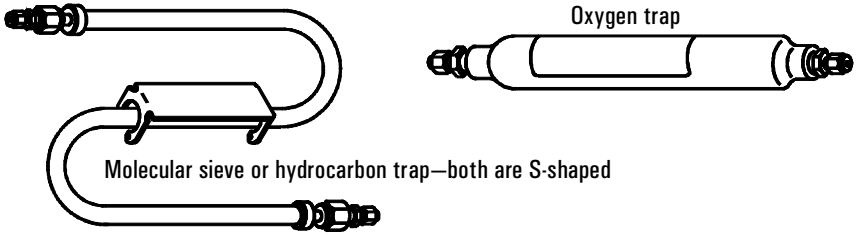


Figure 96. Traps

Cryogenic cooling requirements

Cryogenic cooling allows you to cool the oven below ambient temperature. A solenoid valve introduces liquid coolant, either carbon dioxide (CO_2) or nitrogen (N_2), to cool the oven to the desired temperature.

CO_2 and N_2 require different hardware. You must replace the entire valve assembly if you want to change coolants. The liquid CO_2 valve kit is part no. G1565-65510 and the liquid N_2 kit is part no. G1566-65517.

Choosing a coolant

When selecting a coolant, consider these points:

- The lowest temperature you need to reach
- How frequently you will use cryogenic cooling
- The availability and price of coolant
- The size of the tanks in relation to the size of the laboratory
- Liquid N_2 cools reliably to –80°C
- Liquid CO_2 cools reliably to –40°C

CO_2 is the choice for *infrequent* cryogenic cooling because it does not evaporate and is less expensive than N_2. However, a tank of CO_2 contains much less coolant than a tank of N_2 and more CO_2 is used for the same amount of cooling.

Although liquid N_2 evaporates from the tank regardless of frequency of use, N_2 tanks contain more coolant than do CO_2 tanks and therefore may be better for frequent use.

Using carbon dioxide

WARNING Pressurized liquid CO_2 is a hazardous material. Take precautions to protect personnel from high pressures and low temperatures. CO_2 in high concentrations is toxic to humans; take precautions to prevent hazardous concentrations. Consult your local supplier for recommended safety precautions and delivery system design.

Caution Liquid CO_2 should not be used as a coolant for temperatures below –40°C because the expanding liquid may form solid CO_2—dry ice—in the GC oven. If dry ice builds up in the oven, it can seriously damage the GC.

Liquid CO_2 is available in high-pressure tanks containing 50 pounds of liquid. The CO_2 should be free of particulate material, oil, and other contaminants. These contaminants could clog the expansion orifice or affect the proper operation of the GC.

Additional requirements for the liquid CO_2 system include:

- The tank must have an internal dip tube or eductor tube to deliver liquid CO_2 instead of gas (see Figure 97).
- The liquid CO_2 must be provided to the GC at a pressure of 700 to 1,000 psi at a temperature of 25°C.
- Use 1/8-inch diameter heavy-wall stainless steel tubing for supply tubing. The tubing should be between 5 to 50 feet long.
- Coil and fasten the ends of the tubing to prevent it from “whipping” if it breaks.
- Do not install a pressure regulator on the CO_2 tank, as vaporization and cooling would occur in the regulator instead of the oven.
- Do not use a padded tank (one to which another gas is added to increase the pressure).

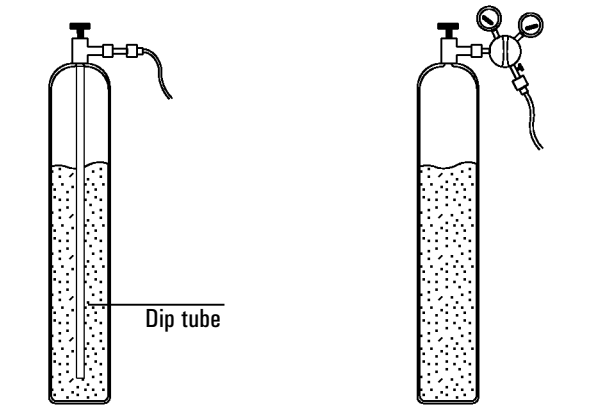


Figure 97. Correct and incorrect liquid CO_2 tank configuration

WARNING Do not use copper tubing or thin-wall stainless steel tubing with liquid CO_2. Both harden at stress points and may explode.

Using liquid nitrogen

WARNING Liquid nitrogen is a hazard because of the extremely low temperatures and high pressures that may occur in improperly designed supply systems.

Liquid nitrogen can present an asphyxiant hazard if vaporizing nitrogen displaces oxygen in the air. Consult local suppliers for safety precautions and design information.

Liquid nitrogen is supplied in insulated Dewar tanks. The correct type for cooling purposes is a *low-pressure* Dewar equipped with a dip tube—to deliver liquid rather than gas—and a safety relief valve to prevent pressure build-up. The relief valve is set by the supplier at 20 to 25 psi.

WARNING If liquid nitrogen is trapped between a closed tank valve and the cryo valve on the GC, tremendous pressure will develop and may cause an explosion. For this reason, keep the delivery valve on the tank open so that the entire system is protected by the pressure relief valve.

To move or replace a tank, close the delivery valve and carefully disconnect the line at either end to let residual nitrogen escape.

Additional requirements for the liquid N_2 system include:

- Nitrogen must be provided to the GC as a liquid at 20 to 30 psi.
- The supply tubing for liquid N_2 must be *insulated*. Foam tubing used for refrigeration and air-conditioning lines is suitable for insulation. Since pressures are low, *insulated* copper tubing is adequate.
- The liquid nitrogen tank should be close (only 5 to 10 feet) to the GC to insure that liquid, not gas, is supplied to the inlet.

Supplying valve actuator air

Some valves use pressurized air for actuation (others are electrically or manually driven). Actuator air must be free of oil, moisture, and particulates. It can be supplied from a dried regulated cylinder, although “house” air supplies or air from a compressor are acceptable.

Most valves require 20 to 40 psi of pressure to operate. High-pressure valves may require 65 to 70 psi.

Valves require a dedicated air supply. Do not share valve air supplies with detectors.

See ”Valve Control”.

29 Installation

Step 1. Unpacking the GC

Step 2. Placing the GC system on the benchtop

Step 3. Turning the power on

Step 4. Connecting tubing to the gas supply tank

Step 5. Attaching traps to the gas supply tubing

Step 6. Attaching a SWAGELOK™ Tee to tubing

Step 7. Attaching tubing to the inlet manifold

Step 8. Attaching tubing to detector manifolds

6890 with Electronic Pressure Control

Step 9. Checking for leaks

Step 10. Attaching cryogenic liquid supplies

Attaching liquid carbon dioxide

Attaching liquid nitrogen

Step 11. Attaching valve actuator air

Step 12. Setting source pressures

Step 13. Connecting cables

Cable diagrams

Analog cable, general use

Remote start/stop cable

Binary-coded decimal cable

External event cable

Step 14. Configuring the GC

Procedure: Setting up a LAN configuration

Installation at a glance

Tools and supplies for installation

Make sure you have the tools and supplies you need before starting the installation.

Wrenches

- ❒ One 5/16-inch
- ❒ One 3/8-inch
- ❒ Two 7/16-inch
- ❒ One 9/16-inch

Screwdrivers

- ❒ T-10 Torx screwdriver
- ❒ T-20 Torx screwdriver

Tubing

- ❒ Copper tubing, 1/8-inch diameter (1/4-inch diameter if $>$ 15 feet (4.6 m) long)
- ❒ Heavy wall, 1/8-inch diameter stainless steel tubing (for liquid CO_2)
- ❒ Insulated copper tubing, 1/4-inch diameter, (for liquid N_2)
- ❒ Tubing cutter

Fittings

- ❒ 1/8-inch SWAGELOK fittings
- ❒ 1/4-inch SWAGELOK fittings (for liquid nitrogen and valve actuator air tubing)
- ❒ 1/8-inch SWAGELOK Tees
- ❒ Nuts and ferrules

Traps (optional)

- ❒ Preconditioned molecular Sieve 5A moisture trap
- ❒ Hydrocarbon trap
- ❒ Oxygen trap

Other

- ❒ Small, flat-blade screwdriver
- ❒ High-quality electronic leak detector
- ❒ Insulating material (for liquid nitrogen tubing only)

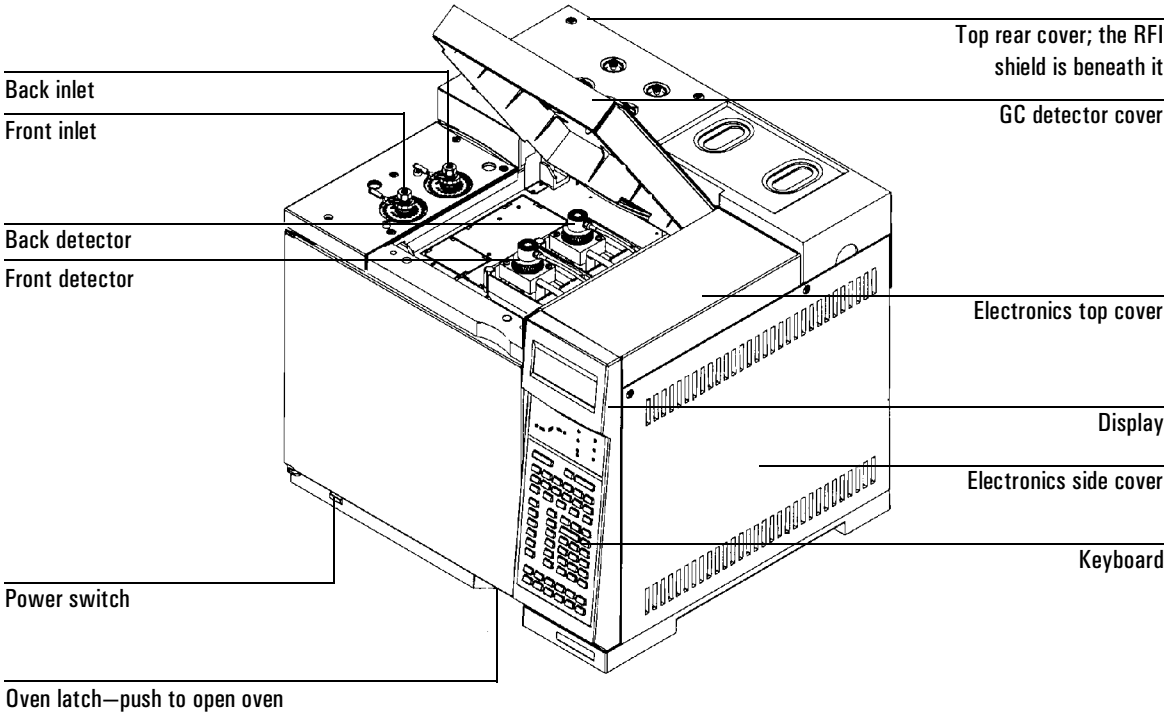


Figure 98. Front view of GC

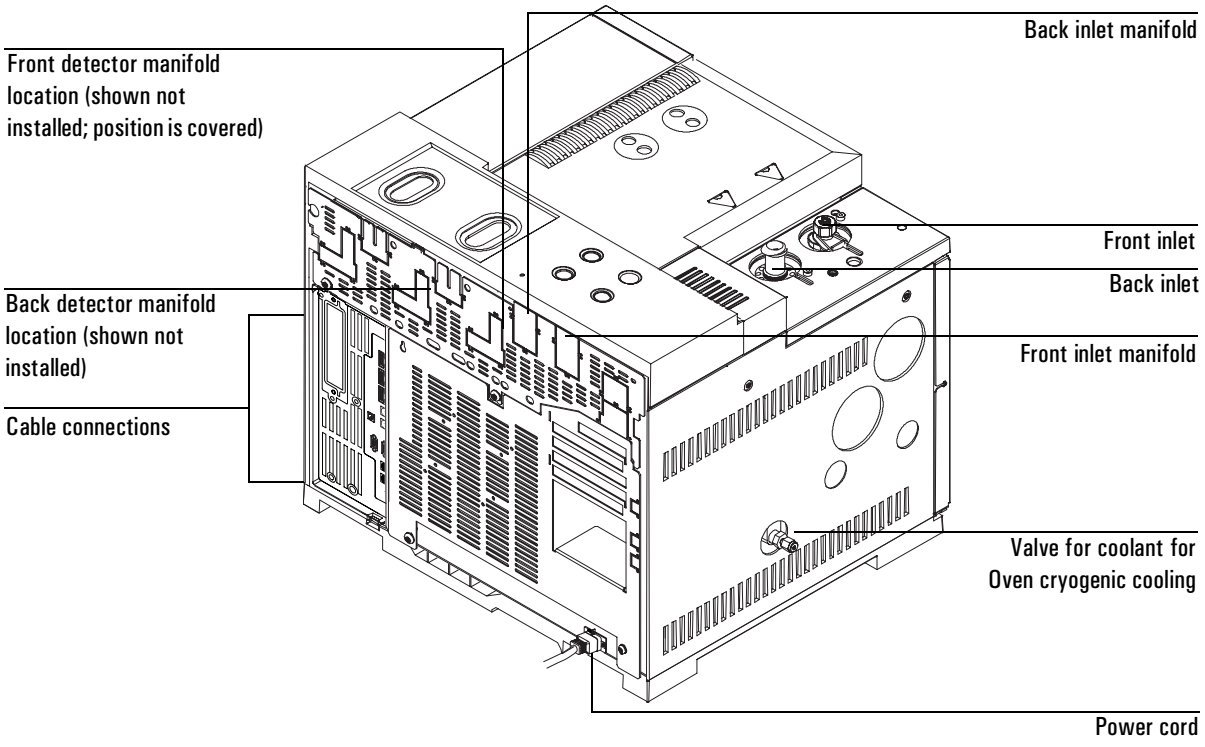


Figure 99. Rear view of GC

Installation

This chapter contains installation procedures for the GC. Most of the installation steps apply to all GC systems—some are optional, such as plumbing for cryogenic cooling and valve actuator air. Instructions are provided for connecting cables from the GC to other instruments in a typical 6890 system. In addition, information about configuring the GC and other instruments is provided.

Most of installation involves plumbing gas to tanks, traps, and manifolds. SWAGELOK™ fittings are used to make leak-tight connections. If you are not sure how to make a SWAGELOK connection, see "Making SWAGELOK Connections" for instruction.

The installation steps assume you need less than 15 feet (4.6 m) of 1/8-inch gas supply tubing for each gas source. For longer installations, use 1/4-inch tubing and appropriate hardware and reducer fittings.

WARNING Hydrogen is a flammable gas. If hydrogen or any other flammable gas is used, periodic leak tests should be performed. Be sure that the hydrogen supply is off until all connections are made, and insure that the inlet fittings are either connected to a column or capped at all times when hydrogen gas is present in the instrument.

Substituting parts or performing any unauthorized modification to the instrument may result in a safety hazard.

The insulation around the inlets, detectors, valve box, and the insulation cups is made of refractory ceramic fibers (RCF). To avoid inhaling RCF particles, we recommend these 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 RCFs.

Step 1. Unpacking the GC

1. Inspect the shipping containers for damage. If a container is damaged or shows signs of stress, notify both the carrier and your local Agilent office.

   Keep all shipping materials for inspection by the carrier.

2. Check the items received against the packing lists. If there are discrepancies, notify your local Agilent office immediately.

   Keep the shipping containers until you have checked their contents for completeness and verified instrument performance.

Step 2. Placing the GC system on the benchtop

The GC requires a benchtop that can support its weight plus that of other equipment you will use with it. Table 70 on page 675 lists some typical weight data. The area must be free of overhanging obstructions that might interfere with cooling and limit access to the top of the instrument.

WARNING Be careful when lifting the GC. Because it is heavy, two people should lift it. When moving the GC, be aware that the back is heavier than the front.

Materials needed:

- ❒ Oven exhaust deflector, part no. 19247-60510 (optional)

1. Remove the GC from its shipping box.
2. Place the GC on the benchtop. Make sure gas and power supplies are accessible. Place other pieces of equipment near the GC as appropriate. See Table 70 on page 675 for suggested benchtop layouts.
3. If space is limited, attach the oven exhaust deflector to the back of the GC as shown below. The deflector hangs from the exhaust vents on four hooks.

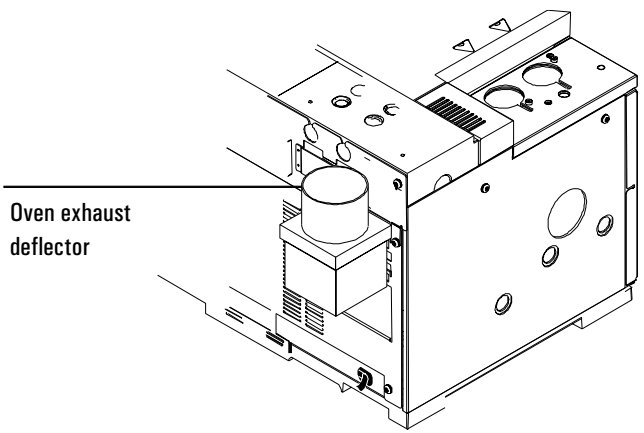


Figure 100. Correct position of the oven exhaust deflector

Step 3. Turning the power on

When you turn the GC on, it runs a series of self-test diagnostics. Run the diagnostics before continuing with the installation to be sure that the instrument electronics are working properly.

1. Verify that the power switch is in the off position.

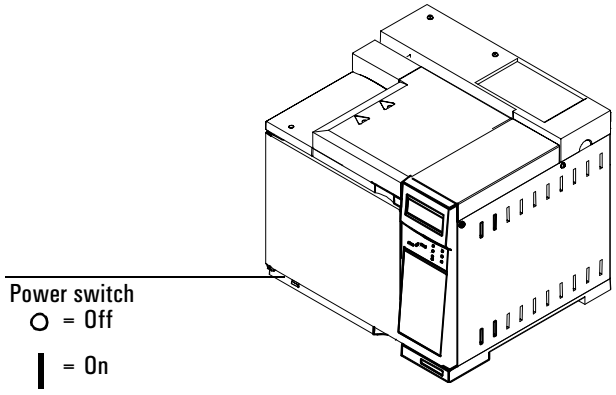


Figure 101. Power switch location

2. Plug the power cord into the back of the GC and power supply. Turn GC on.

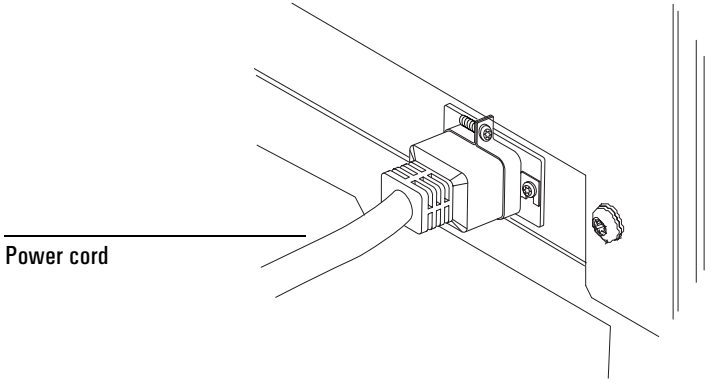


Figure 102. Power cord location

3. The self-test diagnostic tests run automatically. To see the pass/fail message, wait for the test to end and press
   [Oven] [Temp] [On]

   If the screen displays `Power on successful`, turn the GC off and continue with the installation procedure.

   If you do not see this message, turn the GC off and call Agilent service.

Step 4. Connecting tubing to the gas supply tank

Materials needed

- ❒ 1/8-inch preconditioned copper tubing
- ❒ Tubing cutter (part no. 8710-1709)
- ❒ 1/8-inch SWAGELOK nuts, front and back ferrules
- ❒ Two 7/16-inch wrenches

1. Turn off all gases at the source. Determine the length of tubing you need to reach from the gas supply outlet to the inlet manifold on the GC. Take into account any traps or Tee connections you will need.
2. Cut the tubing to length, preferably using a tubing cutter.

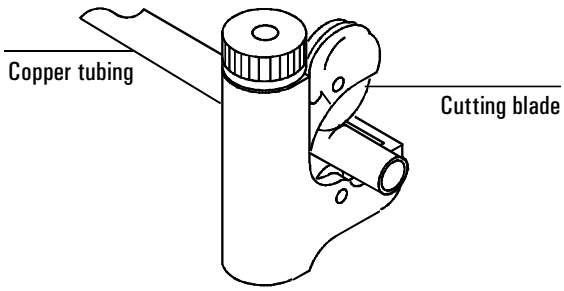


Figure 103. Tubing cutter

3. Connect the tubing to the gas outlet with a SWAGELOK fitting. See "Making SWAGELOK Connections" for information on making SWAGELOK connections.

Step 5. Attaching traps to the gas supply tubing

Materials needed:

- ❒ 1/8-inch preconditioned copper tubing
- ❒ Tubing cutter
- ❒ 1/8-inch SWAGELOK fittings, nuts, and ferrules
- ❒ Two 7/16-inch wrenches and one 1/2-inch wrench
- ❒ Traps

1. Determine where you will install the trap in your supply tubing line. See Figure 104 for the recommended trap order.

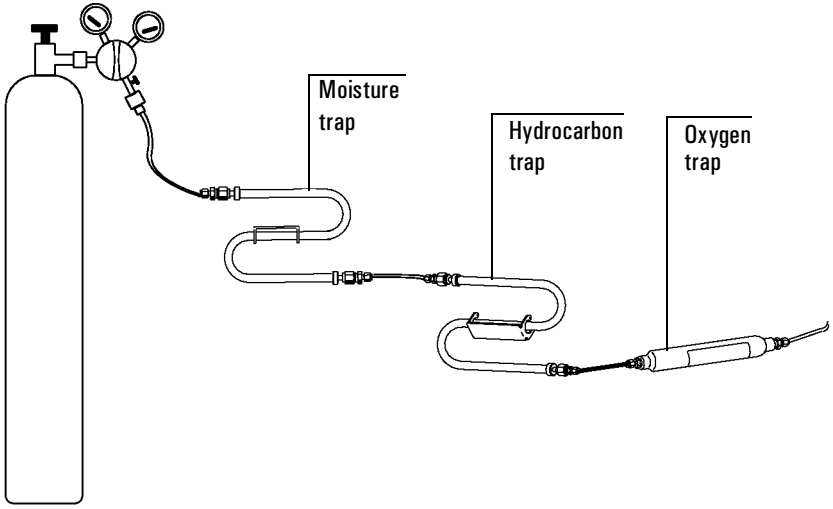


Figure 104. Plumbing diagram

2. Cut the tubing to length using a tubing cutter.
3. Connect the traps and tubing.

Step 6. Attaching a SWAGELOK™ Tee to tubing

If you need to supply gas to more than one inlet or detector module from a single source, use a SWAGELOK ™ Tee near the inlet or detector manifolds.

Materials needed:

- ❒ 1/8-inch preconditioned copper tubing
- ❒ Tubing cutter
- ❒ 1/8-inch SWAGELOK nuts and front and back ferrules
- ❒ 1/8-inch SWAGELOK Tee
- ❒ Two 7/16-inch wrenches
- ❒ 1/8-inch SWAGELOK cap

1. Cut the tubing where you want to install the Tee. Connect the tubing and Tee with a SWAGELOK fitting. See Figure 105.

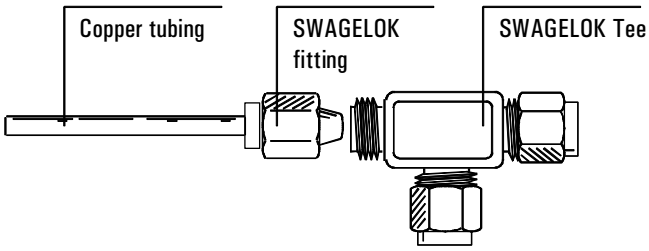


Figure 105. Attaching a SWAGELOK Tee

2. Measure the distance from the Tee to the GC inlets and then attach copper tubing to the open Tee ends with SWAGELOK fittings.
3. You can install a SWAGELOK cap to the open end of a Tee if you do not plan to connect tubing to it immediately.

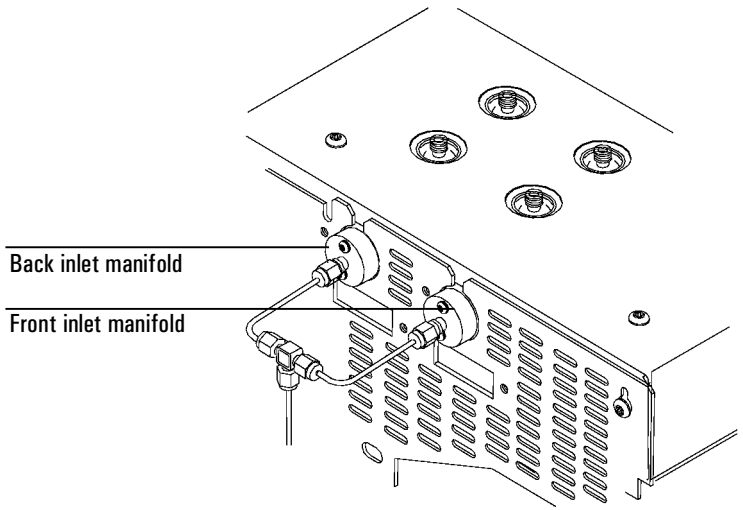
Step 7. Attaching tubing to the inlet manifold

If your GC has EPC inlets, attach the tubing for the gas supply to the inlets on the manifolds on the rear of the instrument. Plumbing for the non-EPC inlets connects inside the pneumatics carrier on the left side of the GC.

Materials needed:

- ❒ 1/8-inch preconditioned copper tubing
- ❒ 1/8-inch SWAGELOK nuts and front and back ferrules
- ❒ Two 7/16-inch wrenches

1. Turn the carrier gas off at its source.
2. Connect the gas supply tubing to the inlet carrier gas manifold with a SWAGELOK nut. See Figure 106.



The GC in this figure has the front and back inlets plumbed with the same carrier gas.

Figure 106. Plumbing the inlet manifolds

Step 8. Attaching tubing to detector manifolds

The gases you connect to a detector depend on the type of detector. The manifolds clearly indicate what types of gas the detectors require and where you should attach the tubing. See the tables on page 680 and for alternative gases for the detector.

This procedure explains how to install gases to the FID. Gases are plumbed to all the detectors in a similar way.

6890 with Electronic Pressure Control

The detector gas inlet fittings are accessible on the instrument back panel.

1. Turn off the gas supplies to be connected at their sources.
2. Each detector gas fitting is labeled. Connect the tubing to the appropriate fitting using a SWAGELOK nut.

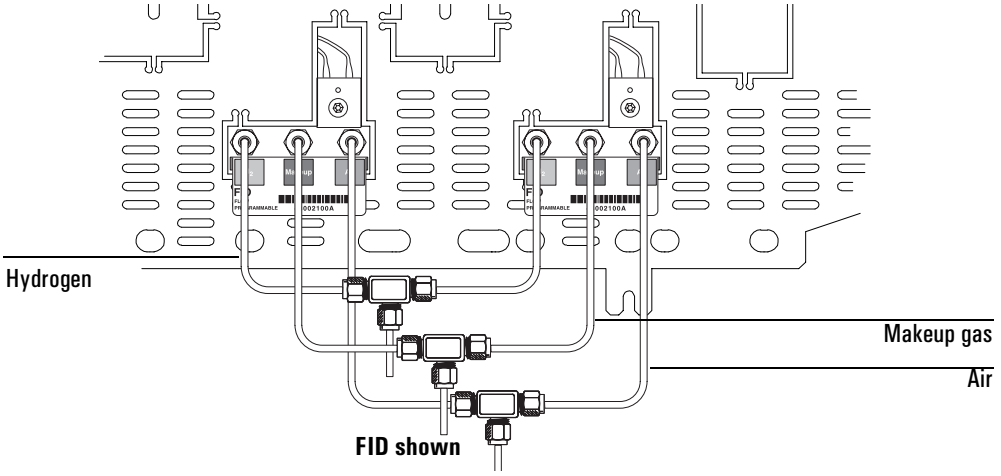


Figure 107. Connecting tubing to an EPC detector

Materials needed:

- ❒ 1/8-inch preconditioned copper tubing
- ❒ Three 1/8-inch SWAGELOK nuts and back and front ferrules sets
- ❒ Two 7/16-inch wrenches

Step 9. Checking for leaks

Liquid leak detectors (Snoop is a common one) are not recommended, especially in areas where cleanliness is very important. If you do use leak detection fluid, immediately rinse the fluid off to remove the soapy film.

WARNING To avoid a potential shock hazard when using liquid detection fluid, turn the GC off and disconnect the main power cord. Be careful not to spill leak solution on electrical leads.

Materials needed:

- ❒ Electronic leak detector (preferred)
- ❒ Leak detection fluid

1. Set the carrier gas pressure at the source (usually tank) regulator to approximately 50 psi.
2. Set the detector gas pressures to the following:
   - Makeup = 50 psi
   - Hydrogen = 50 psi
   - Air = 50 psi
   - TCD reference gas = 50 psi
3. Using the leak detector, check each fitting for leaks.
4. Correct leaks by tightening the connections. Retest the connections; continue tightening until all connections are leak-free.
5. Turn off the inlet and detector gases at the initial supply.

Step 10. Attaching cryogenic liquid supplies

Cryogenic cooling allows you to operate the GC below ambient temperature. A solenoid valve introduces liquid coolant, either CO_2 or N_2, at a rate appropriate to cool the oven to the desired temperature.

The choice of coolant depends largely on how frequently you use cryogenic cooling. You cannot use CO_2 and N_2 interchangeably because they require different valve assemblies. For more information on choosing cryogenic coolant, see "Cryogenic cooling requirements".

Flared or AN tubing fittings are commonly used to connect the liquid supply tubing to the cryo coolant tank. Check with the supplier of the coolant before plumbing to be sure you have the correct fittings.

Attaching liquid carbon dioxide

WARNING Do not use copper or thin-wall stainless steel tubing! Either presents an explosion hazard.

Caution Do not use padded tanks for CO_2 supplies. The cryogenic valve is not designed to handle the higher pressures padded tanks generate.

Materials needed:

- ❒ 1/8-inch heavy-wall, stainless steel tubing
- ❒ Tubing cutter
- ❒ 1/8-inch SWAGELOK nuts and ferrules
- ❒ Two 7/16-inch wrenches

1. Locate the inlet for liquid CO_2 on the **left** side of the GC. Prepare enough tubing to reach from the supply tank to this fitting. See Figure 108.

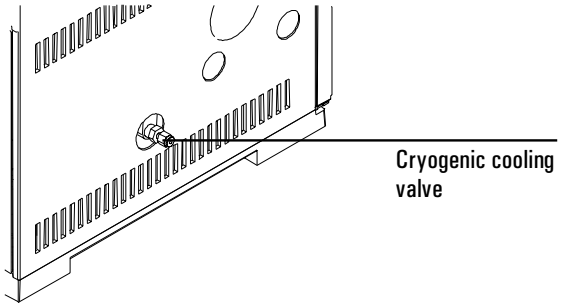


Figure 108. Location of cryogenic cooling valve

2. Connect the supply tubing to the liquid CO_2 tanks outlet with the fitting recommended by the supplier.
3. Use a SWAGELOK fitting to connect the supply tubing to the cryogenic valve inlet.

Attaching liquid nitrogen

Materials needed:

- ❒ 1/4-inch insulated copper tubing
- ❒ Tubing cutter
- ❒ 1/4-inch SWAGELOK fittings, nuts, and ferrules
- ❒ Two 9/16-inch wrenches

1. Position the nitrogen tank as close to the GC as possible to insure that liquid and not gas is delivered to the inlet.
2. Locate the inlet for coolant on the left-hand side of the GC. Prepare enough tubing to reach from the supply tank to this outlet. See Figure 109.

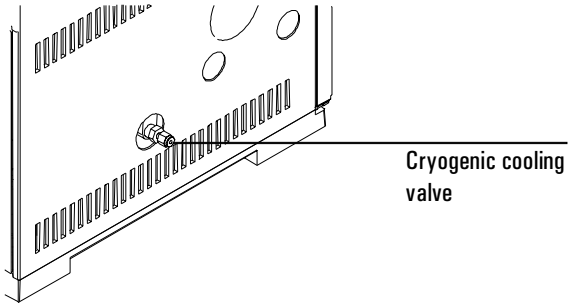


Figure 109. Location of cryogenic cooling valve

3. Connect the supply tubing to the liquid N_2 tank outlet with the fitting recommended by the supplier.
4. Use a SWAGELOK fitting to connect the supply tubing to the cryogenic valve inlet.

Step 11. Attaching valve actuator air

Valves require air to actuate. Valves should have a dedicated air source; they cannot share detector air supplies.

Valve actuator air is supplied through 1/4-inch plastic tubing. If your GC has valves, the plastic tubing will already be attached to the actuators and will extend from the back of the GC.

Caution Route the tubing away from the oven exhaust. The hot air will melt the plastic tubing.

Materials needed:

- ❒ 1/4-inch SWAGELOK fittings and front and back ferrule
- ❒ Two 9/16-inch wrenches

Turn the air off at the source. Use a sharp knife if you need to shorten the tubing. Connect the tubing to the air source using a 1/4-inch SWAGELOK nut. See Figure 110.

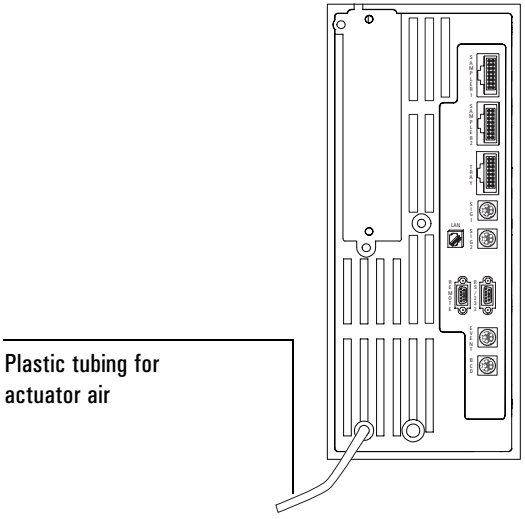


Figure 110. Location of valve actuator air tubing

Step 12. Setting source pressures

The pressure set at a tank regulator depends on these factors:

- The pressure needed to achieve the highest flow rate you intend to use.
  The pressure/flow relationship depends on the column or device involved. The best way to address this is to begin at a moderate pressure level and adjust upward as needed.
- A pressure difference of about 10 psi (138 kPa) across pressure and flow sensing and controlling devices to enable them to work properly.
  This pressure difference requirement is much the same for all sensors and controllers, including flow controllers and pressure regulators.
- The pressure limit of the *weakest* part of the supply system.
  Swagelok fittings and copper tubing are more than adequate for the highest gas pressures encountered in gas chromatography.

  The pneumatics modules of the GC will withstand over 250 psi pressure, but may not function reliably. We recommend a maximum continuous operating pressure of 170 psi to avoid excessive wear and leaks.

  Traps are often the weakest part of the system. They should be labeled, either on the trap itself or in accompanying literature, with a maximum operating pressure. Source pressure must not exceed the *lowest* maximum operating pressure in the supply system.

Suggested starting values of source pressure are:

Gas	Use	Source pressure
Carrier	Packed columns	410 kPa (60 psi)
	Capillary columns	550 kPa (80 psi)
Air	Detectors	550 kPa (80 psi)
Hydrogen	Detectors	410 kPa (60 psi)

Refer to Table 76 for inlet and detector maximum and minimum pressures.

Step 13. Connecting cables

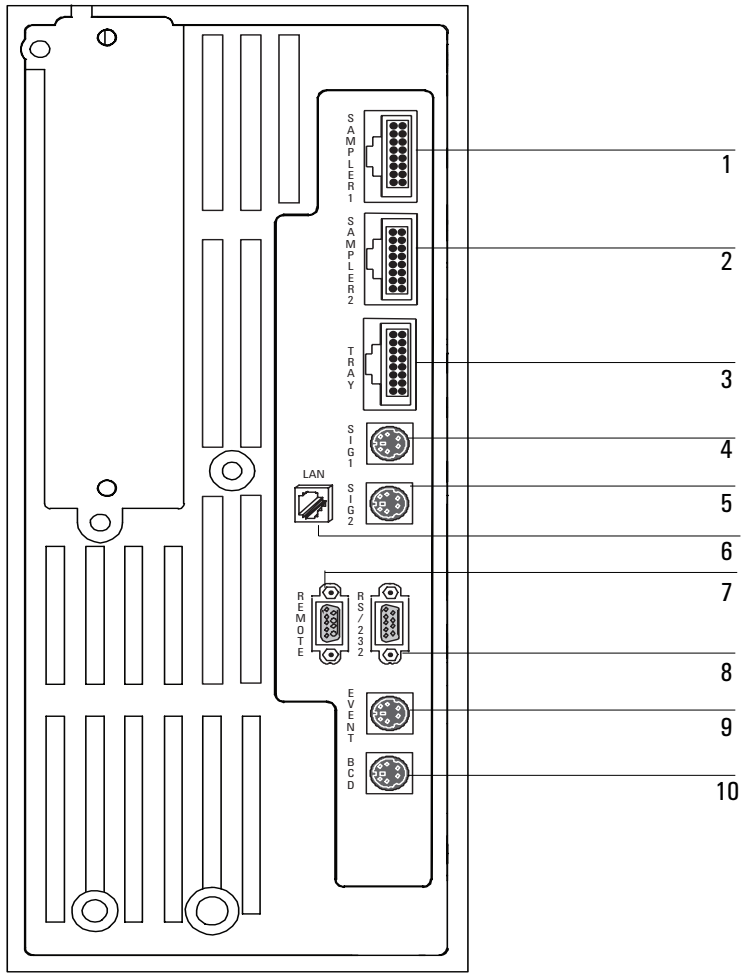
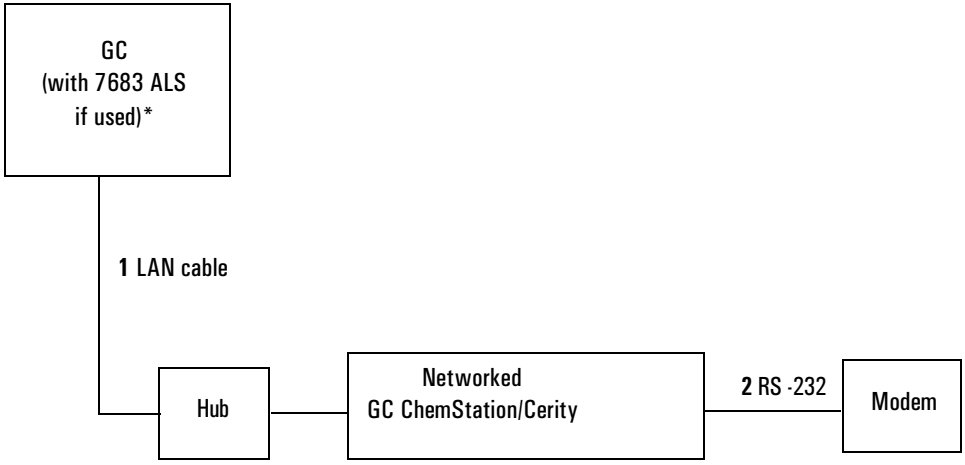


Figure 111. Overview of cable connections on the back of the GC

The GC has an extensive set of communication tools:

- **1, 2 Sampler** Power and communications for a G2613A injector. Use Sampler1 for the front injector.
- **3 Tray** Power and communications for an G2613A tray.
- **4, 5 Analog signal outputs** Two channels of analog data output for use with external signal processors. Each analog output has three voltage ranges.
- **6 LAN** LAN communications
- **7 Remote** Remote port that can be used to synchronize up to ten instruments.
- **8 Modem/RS-232C** For use with modems, computers, and other controller devices.
- **9 External event control** Two passive contact closures and two 24-volt control outputs for controlling external devices. Connected to valve drivers 5 through 8 on the GC.
- **10 BCD** (Binary-Coded Decimal) This connector provides the control relays and a BCD input for a stream selection valve. Does not provide output for use with data handling devices.

There are many system configurations possible with the GC. The figures show two common configurations. See Table 78 and Table 79 for cabling requirements for other combinations. See Figure 112.



* The 7683 controller is internal to the 6890 GC. The G2613A Injector and the 2614 tray plug directly into the GC.

Figure 112. GC—networked GC ChemStation/Cerity—GC Automatic Liquid Sampler

Number	Part no. and description
1	92268 B, LAN cable, Ether twist 4 pair
2	G1530-61120, RS-232/modem cable or 24540-80012, RS-232/modem cable

Table 78. Cabling Requirements

Instrument Connected to	Required Cable(s)	Part no.
7683 Automatic Liquid Sampler	Injector cable is integral Tray cable	 G2614-60610
GC ChemStation	LAN (see below)	
7694 Headspace Sampler	Remote, 9-pin male/6-pin connector	G1290-60570
7695 Purge and Trap Sampler	Remote, 25-pin male/9-pin male	G1500-60820
3395A Integrator	Remote, 9 pin/15 pin Analog, 2 m, 6 pin	03396-61020 G1530-60570
3395B Integrator	Remote, 9 pin/15 pin Analog, 2 m, 6 pin	03396-61010 G1530-60570
3396B Integrator	Remote, 9 pin/15 pin Analog, 2 m, 6 pin	03396-61020 G1530-60570
3396C/3397 Integrator	Remote, 9 pin/15 pin Analog, 2 m, 6 pin	03396-61010 G1530-60570
Non-Agilent Integrator	Analog, 2 m, 6 pin	G1530-60560
35900 C/D/E A/D Converter	Remote, 9-pin male/9-pin male Analog, 2 m, 6 pin	G1530-60930 G1530-60570
Mass Selective Detector	Remote, 2-m, 9-pin male/9-pin male	G1530-60930
Modem	Modem, 9-pin female/9-pin male, or Modem, 9-pin female/25-pin male	G1530-61120, or 24540-80012
Non-Agilent data system	General use remote, 9-pin male/spade lugs External event, 8-pin/spade lugs	35900-60670 (2 m), 35900-60920 (5 m), 35900-60930 (0.5 m) G1530-60590
Non-Agilent instrument, unspecified	External event, 8 pin/spade lugs	G1530-60590
Stream selection valves Gas sampling valves	See documentation accompanying the valve	
LAN	Ether Twist 4 pair	92268B

Table 79. Cabling for Other Instruments in a 6890 System

Instrument 1	Instrument 2	Type of cable	Part no.
Mass Selective Detector	GC ChemStation	LAN	92268B
GC ChemStation	Modem	RS-232	24540-80012, or G1530-61120
7694 Headspace Sampler	GC ChemStation	RS-232, 9-pin female/ 9-pin male	24542U
7694 Headspace Sampler	Integrator	RS-232, 15-pin male/ 9-pin female	03396-60530
7694 Headspace Sampler	Unspecified, non-Agilent instrument	Binary-coded decimal cable	03396-60570
Mass Selective Detector	Purge & Trap or Headspace sampler	Splitter ("Y") cable for remote, 1 male and 2 female connectors	G1530-61200
		Splitter ("H") cable for APG remote, 2 male and 2 female connectors	35900-60800

Cable diagrams

If you connect the GC to a non-Agilent instrument or to the 35900 A-to-D Converter, you must know the function of each wire in the cable. See Table 80.

Analog cable, general use

The GC uses the general use analog cable to communicate with a non-Agilent integrator. The general use cable is also used with non-Agilent detectors. See Figure 113.

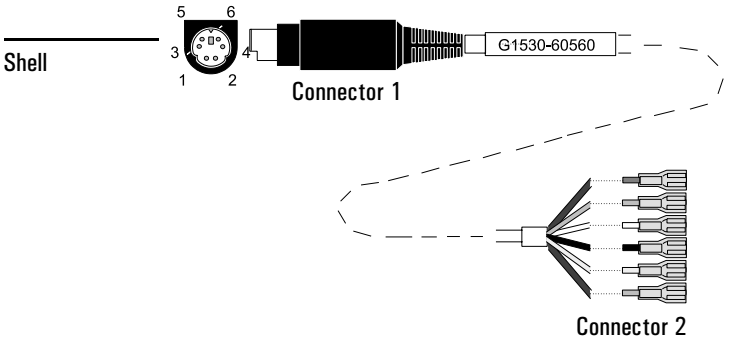


Figure 113. Analog cable, general use (part no. G1530-60560)

Table 80. Analog Cable, General Use Output Connections

Connector 1	Connector 2—Quick connects	Signal
1	Brown or violet	0 to 1 mV (–)
2	White	0 to 1 V, 0 to 10 V(–)
3	Red	0 to 1 mV (+)
4	Black	1 V (+)
6	Blue	10 V (+)
Shell	Orange	Ground

Remote start/stop cable

Two ports are available to remotely start and stop instruments in a loop. For example, you might have an integrator, automatic sampler, and a gas chromatograph connected with Remote cables. You can synchronize a maximum of ten instruments using Remote cables. See Figure 114 and Table 81.

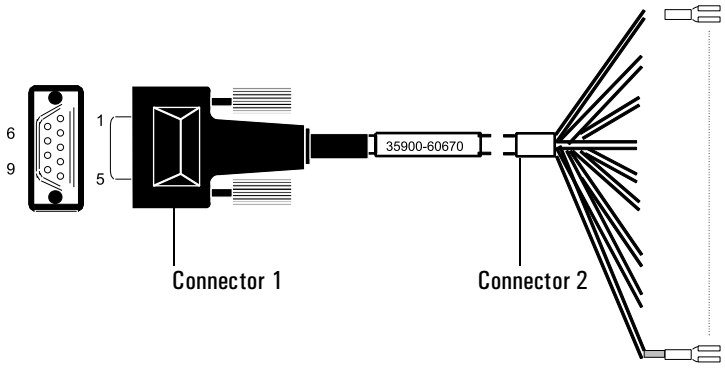


Figure 114. Remote start/stop cables

Table 81. Remote Start/Stop Connections

Connector 1 9 pin male	Connector 2 35900-60670 spade lugs	Signal name
1	Black	Digital ground
2	White	Prepare (low tone)
3	Red	Start (low tone)
4	Green	Start relay (closed during start)
5	Brown	Start relay (closed during start)
6	Blue	Open circuit
7	Orange	Ready (high true input)
8	Yellow	Stop (low tone)
9	Violet	Open circuit

Binary-coded decimal cable

The BCD cable contains eight passive inputs that sense total binary-coded decimal levels. See Figure 115 and Table 82.

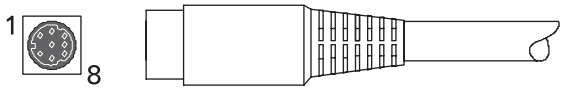


Figure 115. Typical BCD input cable

Table 82. BCD Input Connections

Pin	Function	Maximum rating
1	Relay	48 V AV.DC, 250mA
2	Relay	48 V AC/DC, 250mA
3	LS digit 0	
4	LS digit 1	
5	LS digit 2	
6	LS digit 3	
7	MS digit 0	
8	GND	
Shield	Chassis GND	

External event cable

Two passive relay contact closures and two 24-volt control outputs are available for controlling external devices. Devices connected to the passive contact

closures must be connected to their own power source. See Figure 116 and Table 83.

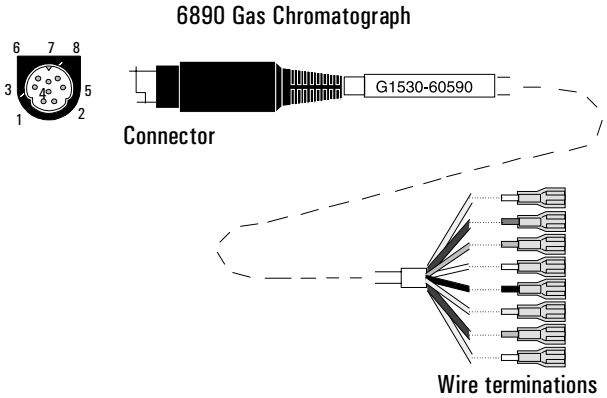


Figure 116. External event cable (part no. G1530-60590)

Table 83. External Event Connections

Connector	Signal name	Maximum rating	Wire terminations	Corresponds to valve #
24 volt control output				
1	24 volt output 1	75 mA output	Yellow	5
2	24 volt output 2	75 mA output	Black	6
3	Ground		Red	
4	Ground		White	
Relay contact closures (normally open)				
5	Contact closure 1	48V AC/DC, 250 mA	Orange	7
6	Contact closure 1		Green	7
7	Contact closure 2	48 V AC/DC, 250 mA	Brown or violet	8
8	Contact closure 2		Blue	8

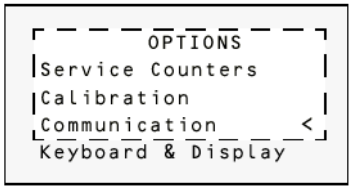
Step 14. Configuring the GC

For network (LAN) operation, you must first configure the GC. You can configure the GC to automatically receive its TCP/IP addressing information from a DHCP name server, or set the address directly at the keyboard.

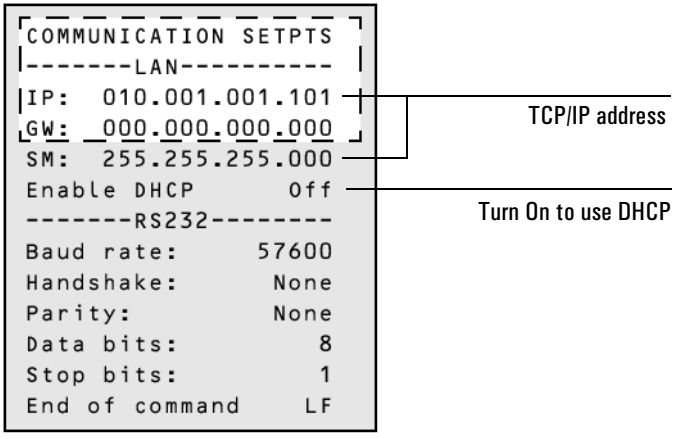
Consult your data system documentation or contact your LAN administrator to determine the appropriate TCP/IP addressing information.

Procedure: Setting up a LAN configuration

1. At the keyboard, press [Options].



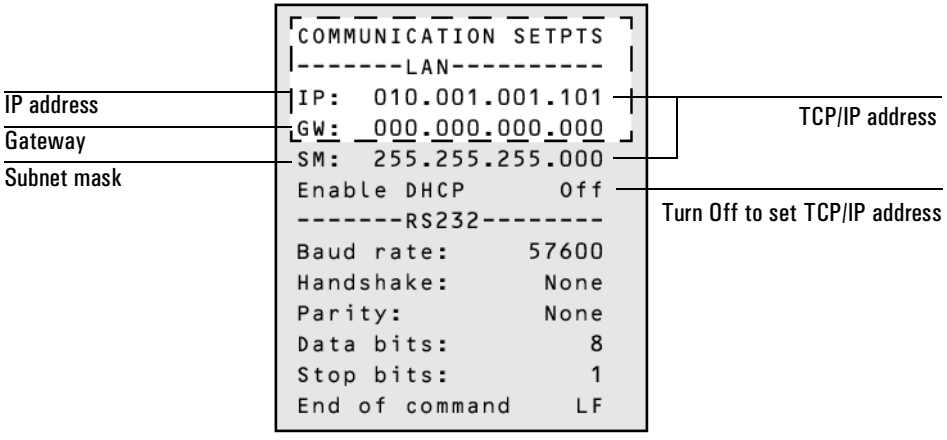
2. From the open control table, scroll to Communication. Press [Enter].
3. **To use DHCP to set the GC's LAN address**, scroll to Enable DHCP and turn it On. The TCP/IP address information disappears. When prompted, turn the GC off then on again to use the new setting.



4. **To set the LAN address at the GC front keyboard**, scroll to Enable DHCP and turn it Off.

   - Enter the TCP/IP address information in the appropriate spaces.
   - Press [Enter] to input each item, or press [Clear] to cancel changes.
   - After each entry, you will be prompted to turn the GC off, then on again. Press any key to clear this message.

   After completing all entries, turn the GC off then on again to use the new setting.



30 Making SWAGELOK Connections

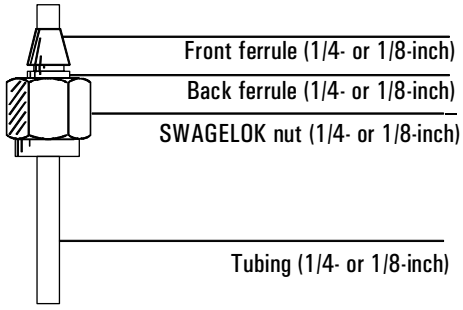
SWAGELOK Connections

The gas supply tubing is attached with SWAGELOK fittings. If you are not familiar with making SWAGELOK connections, review the following procedure. The procedure explains how to connect tubing to a fitting, such as inlet and detector manifolds or the gas supply tank.

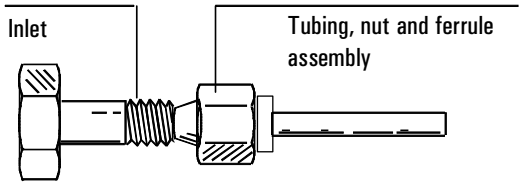
Materials needed:

- ❒ 1/8-inch (or 1/4-inch, if used) preconditioned copper tubing
- ❒ 1/8-inch (or 1/4-inch, if used) SWAGELOK nuts, and front and back ferrules
- ❒ Two 7/16-inch wrenches

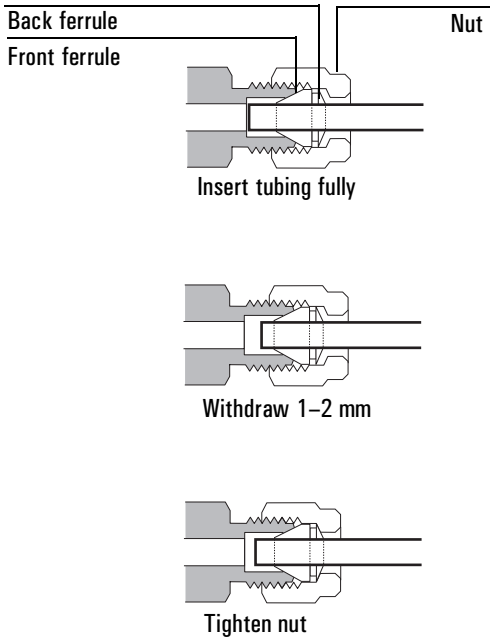
1. Attach a 1/8-inch SWAGELOK nut, back ferrule, and front ferrule to the tubing.



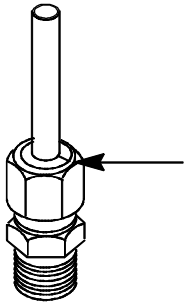
2. Make sure that the front ferrule is touching the inlet, and then slide the SWAGELOK nut over the ferrule and tighten it finger-tight.



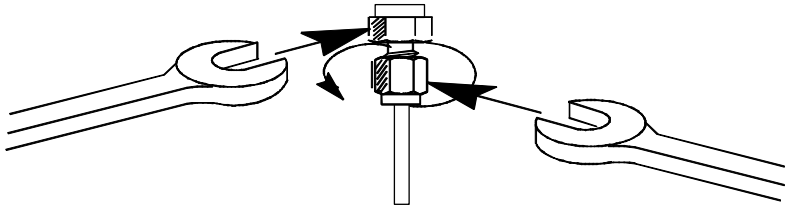
3. Push the tube fully into the female fitting, then withdraw it approximately 1–2 mm.



4. Mark the SWAGELOK fitting with a pencil line.



5. If you are using 1/8-inch SWAGELOK fittings, while holding the fitting steady with the other 7/16-inch wrench, tighten the fitting 3/4 of a turn. If you are using 1/4-inch fittings, tighten them 1 1/4 turn.



Tightening SWAGELOK nuts by this procedure provides a leak-proof, torque-free seal at all tubing connections.

A

Activated charcoal trap
  Description 687
  Installation 703
Actual value 37
Actuator air
  Plumbing 711
  Requirements 692
Adapter
  bubble meter 96
  detector
    installation 147
  PTV inlet, replacing 421
Ambient temp
  oven setpoint 116
Analog
  Cable G1530-60560 718
  Cable G1530-60570 716
  zero 172
Analog output 714
  attenuation 174
  range 173
Analog-to-Digital Converter, 35900 675, 716
APG remote communications
  see Remote start/stop 714
Asterisk 36
Attenuation 174
Auto Prep Run 285
Automatic liquid sampler
  7683
    bar code reader 211
    configuring injector 210
    control table 207
    injector setpoints 208
    sample tray setpoints 211
  Specifications 675
Aux # keys 42
Auxilary temperature programming 215
Auxiliary channels 88

B

Back Det key 42
Back Injector key 60
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