3 Flow and Pressure Control

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

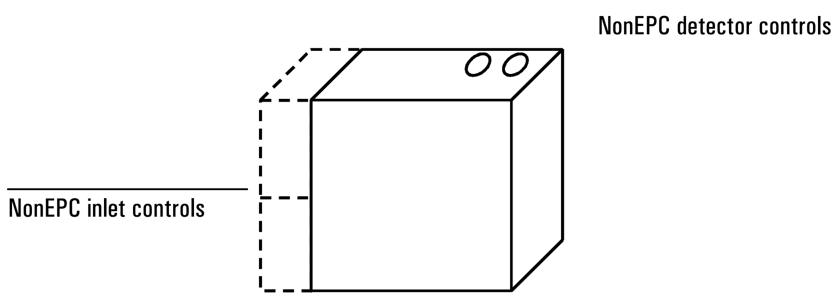


Figure 5 Control location
The pneumatics module (dashed lines) is present if a nonEPC inlet is installed.

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

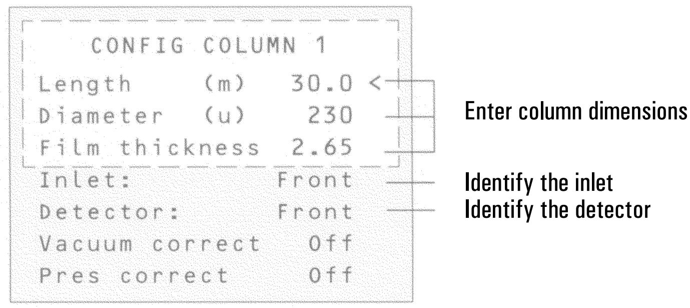
You define (configure) a capillary column by entering its length, diameter, and film thickness. With this information, the instrument can calculate the flow through the column. This has great advantages when using capillary columns because it becomes possible to:

- 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.

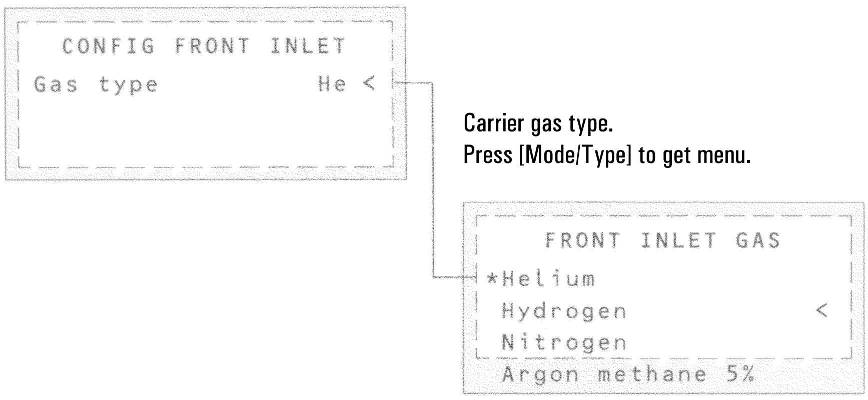
  It is possible, and sometimes appropriate, to configure both installed columns to the same inlet.
- Some pneumatic setpoints change with oven temperature because of changes in column resistance and in gas viscosity. This may confuse users who observe pneumatics setpoints changing when their oven temperature 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

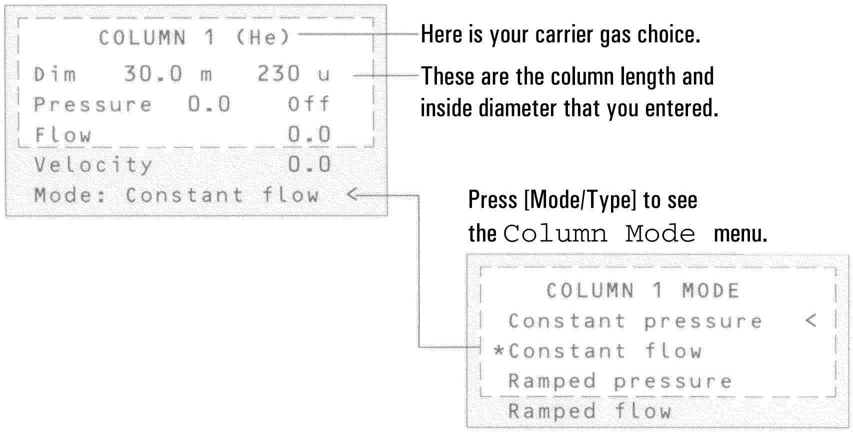
Pressures are gauge pressures—the difference between the absolute pressure and the local atmospheric pressure. Because most detectors present little resistance to the column flow, the gauge pressure at the column head is usually the same as the pressure difference between column inlet and exit. The mass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

```
    COLUMN 1
Dim    50.0 m230 u
Pressure   2.5     2.5
Flow              10.0
Velocity            74
Mode: Constant flow  <
```

- Dim: The column length and inside diameter.
- Pressure, Flow, Velocity: You set one of these. The GC calculates the other two.
- Mode: The column mode; see below.

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

```
Mode: Const flow      <
```

```
Mode: Const pressure <
```

```
Mode: Ramped flow    <
Init flow          4.0
Init time          2.0
Rate 1             0.5
Final flow        18.0
Final time        12.0
Rate 2 (Off)      0.00
```

```
Mode: Ramped pressure<
Init pressure10.0
Init time          1.0
Rate 1             1.0
Final pressure1 25.0
Final time        15.0
Rate 2 (Off)      0.00
```

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`.

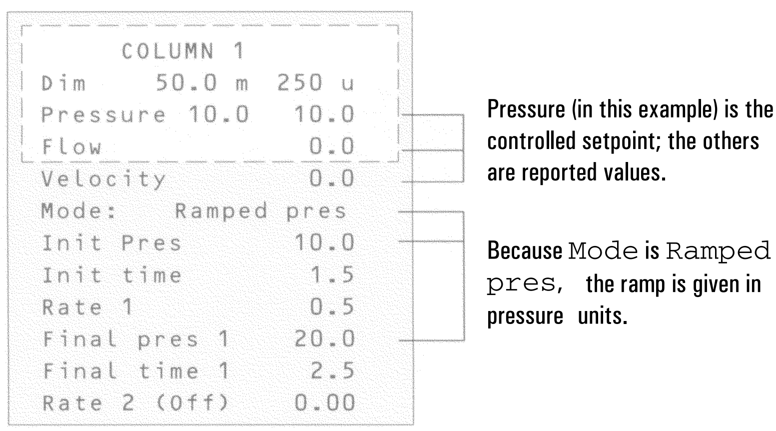
The program ends when it reaches a `Rate` that is set to 0 (off).

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.

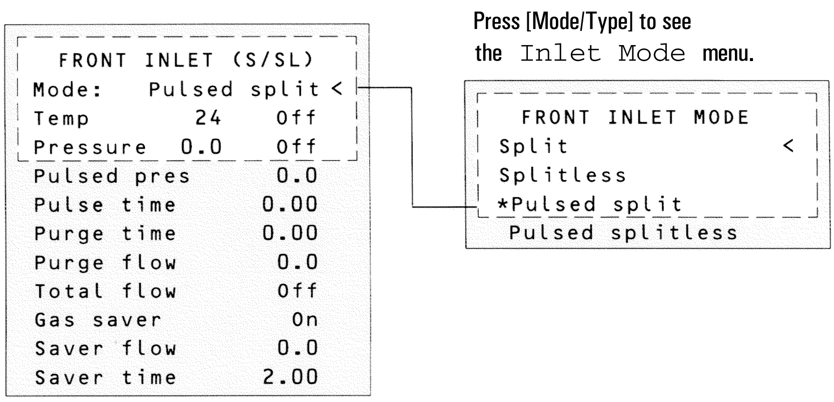
The split/splitless inlet has a gas saver feature. This reduces the flow of carrier into the inlet and out the split vent after the injection is complete. It does not alter the flow through the column.

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"*.

Split mode

Inlet temperature — Temp
Split parameters — Split ratio, Split flow, Tot flow
Gas saver parameters — Gas saver, Saver flow, Saver time

FRONT INLET (S/SL)		
Mode:		Split
Temp	24	Off <
Pressure	0.0	Off
Split ratio		100
Split flow		0.0
Tot flow	0.0	Off
Gas saver		On
Saver flow		0.0
Saver time		2.00

Splitless mode

Splitless parameters — Purge time, Purge flow, Total flow

FRONT INLET (S/SL)		
Mode:	Splitless	
Temp	24	Off <
Pressure	0.0	Off
Purge time		0.00
Purge flow		0.0
Total flow		Off
Gas saver		On
Saver flow		0.0
Saver time		2.00

The pulsed modes

Pulse parameters — Pulsed pres, Pulse time

FRONT INLET (S/SL)		
Mode:	Pulsed Split	
Temp	24	Off <
Pressure	0.0	Off
Pulsed pres		0.0
Pulse time		0.00
Split ratio		100
Split flow		0.0
Tot flow	0.0	Off
Gas saver		On
Saver flow		0.0
Saver time		2.00

FRONT INLET (S/SL)		
Mode:Pulse Splitless		
Temp	24	Off <
Pressure	0.0	Off
Pulsed pres		0.0
Pulse time		0.00
Purge time		0.00
Purge flow		0.0
Total flow		Off
Gas saver		On
Saver flow		0.0
Saver time		2.00

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.

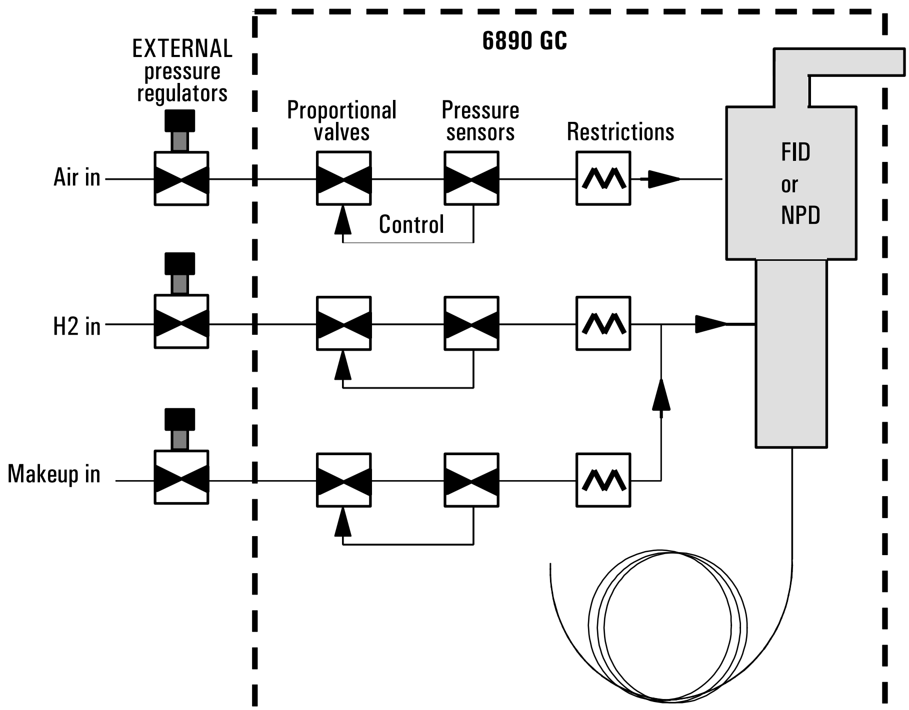


Figure 7 Internal/external plumbing: FID and NPD *with* EPC

For more detail, see "The Flame Ionization Detector", "The Nitrogen-Phosphorus Detector".

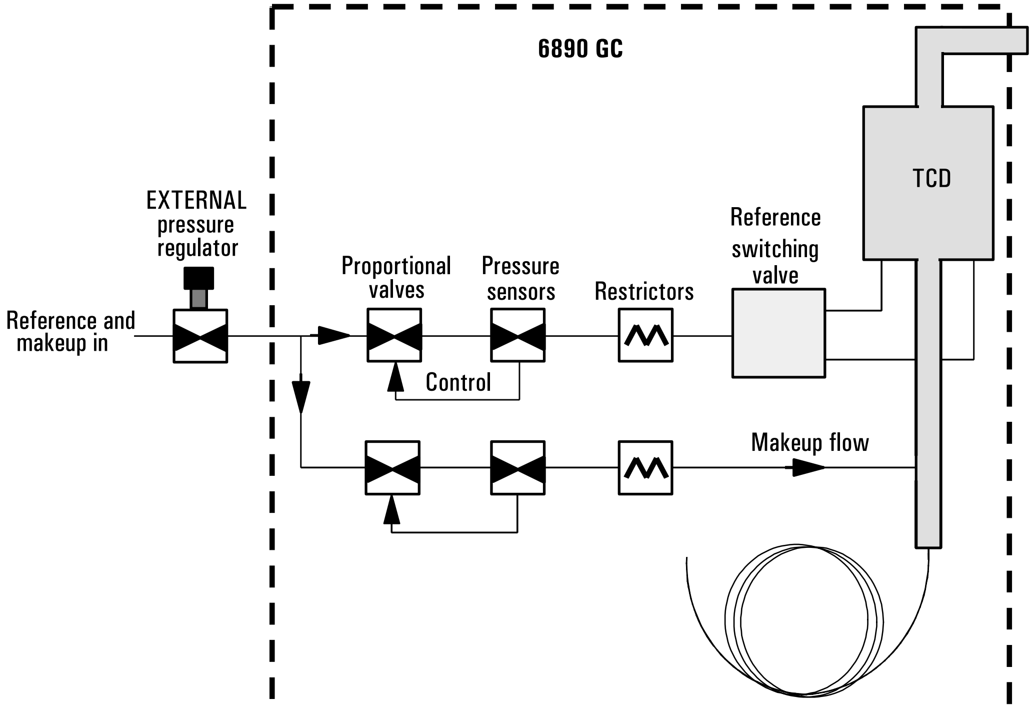


Figure 8 Internal/external plumbing: TCD *with* EPC

For more detail, see "The Thermal Conductivity Detector".

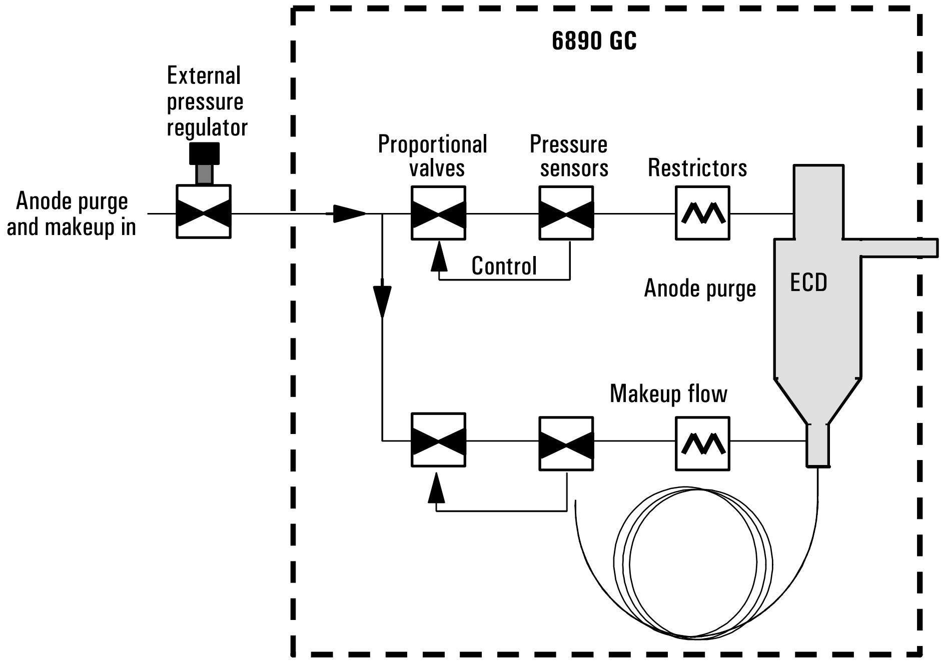


Figure 9 Internal/external plumbing: ECD *with* EPC

For more detail, see "The Micro-Cell Electron Capture Detector".

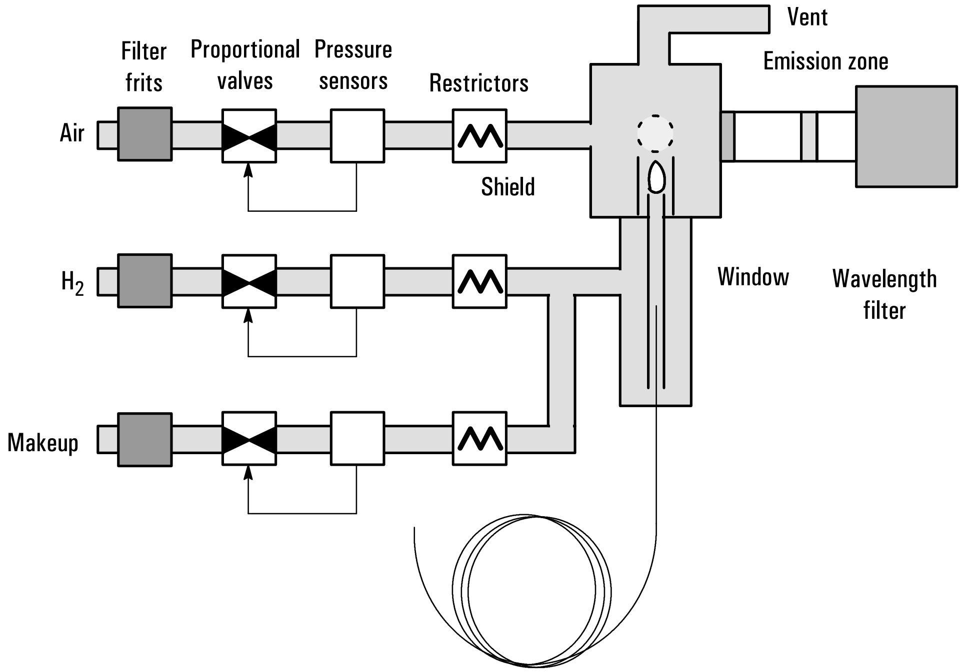


Figure 10 Internal/external plumbing: FPD *with* EPC
For more details, see "The Flame Photometric Detector".

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

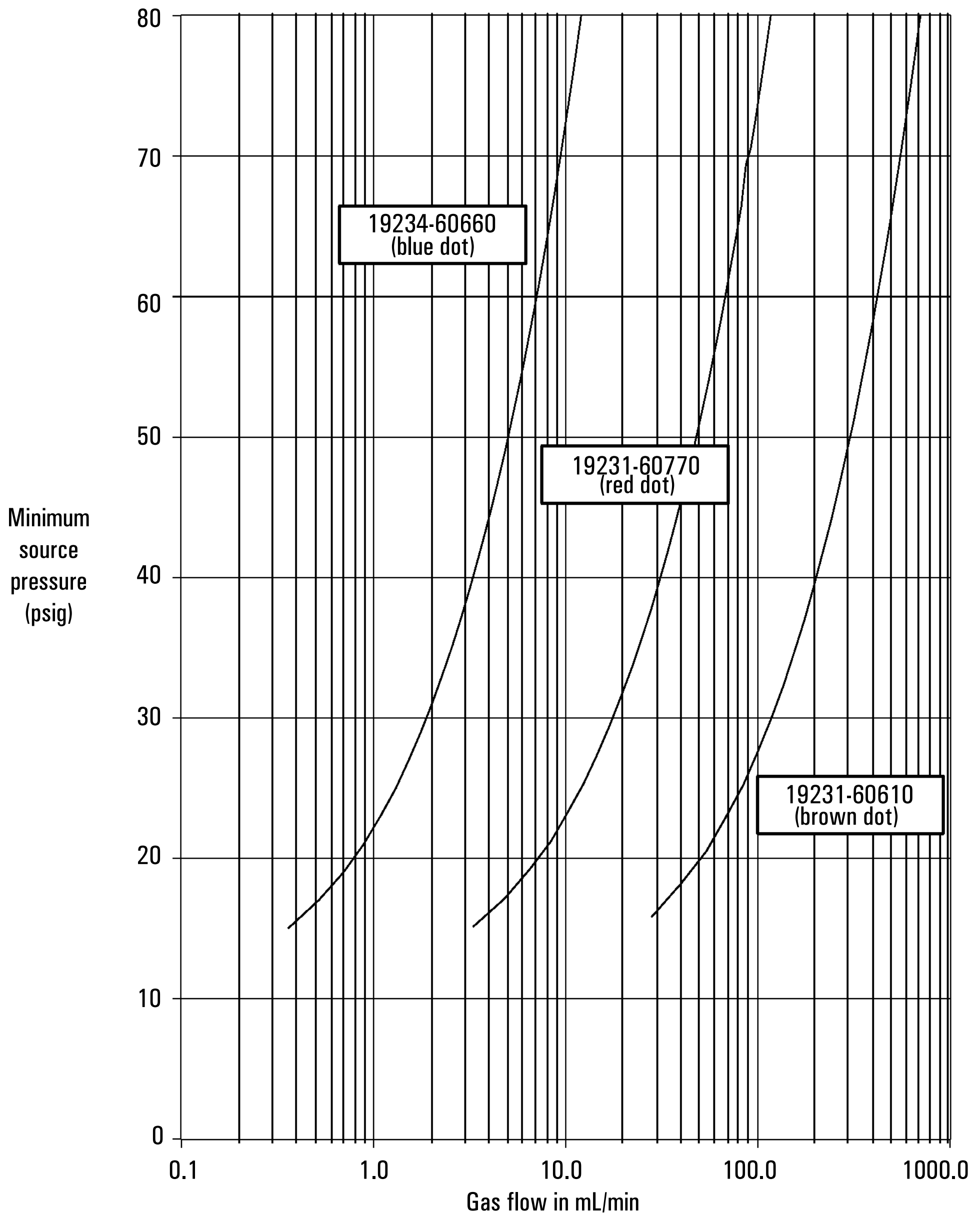
The Red Dot frit is in all three channels when the instrument is shipped.

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.

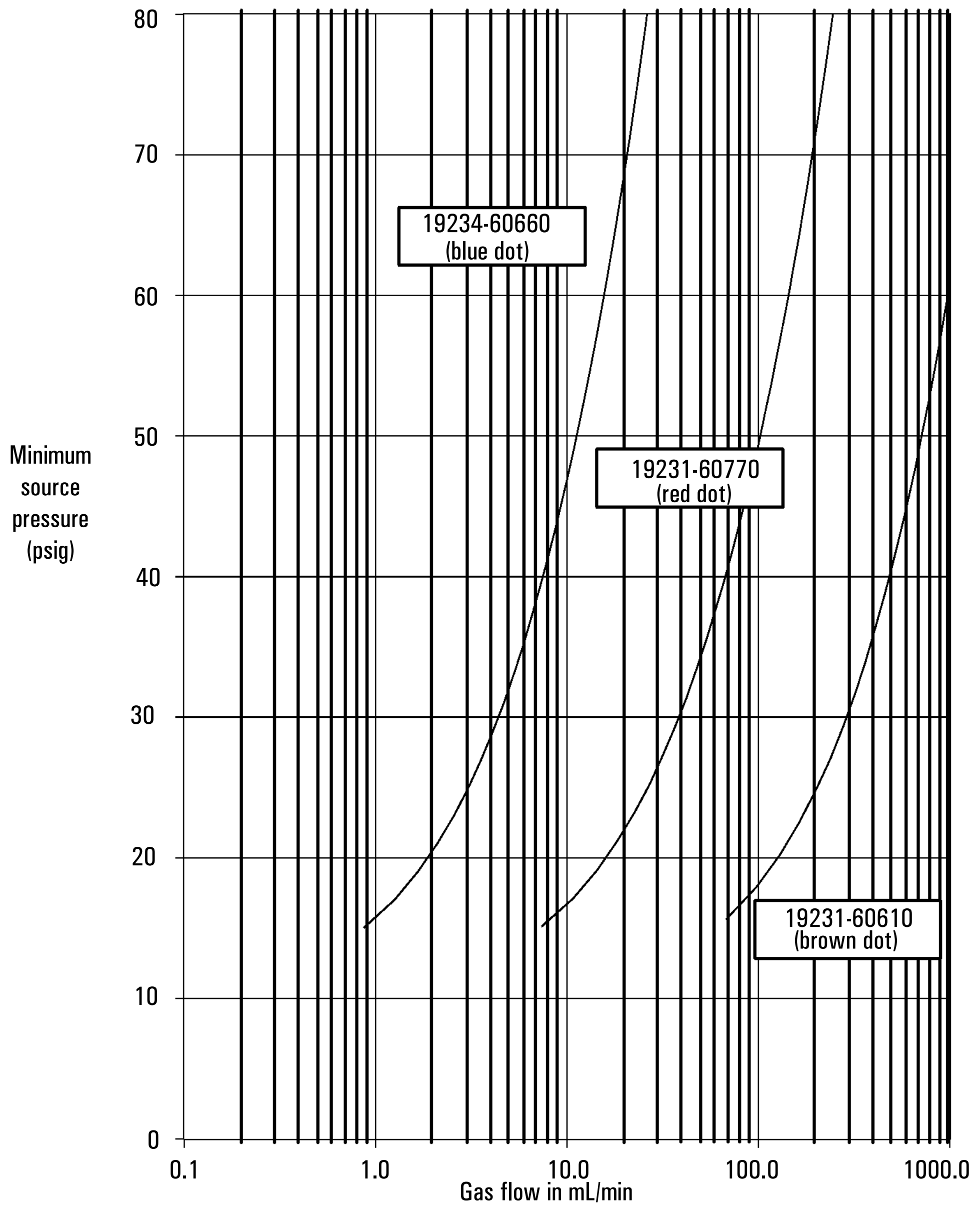
If the Zero resistance frit is installed, the user must provide flow resistance downstream and generate the pressure/flow relationships.

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)

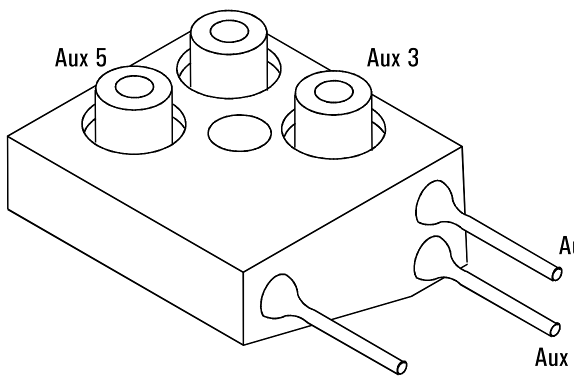


Pressure requirements for AUX EPC flow restrictors
with hydrogen
(ambient conditions: 25°C, 14.7 psia)



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]

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

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

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

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

or

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

For flow:

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.

For pressure:

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

To zero all pressure sensors in all modules

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

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

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

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.

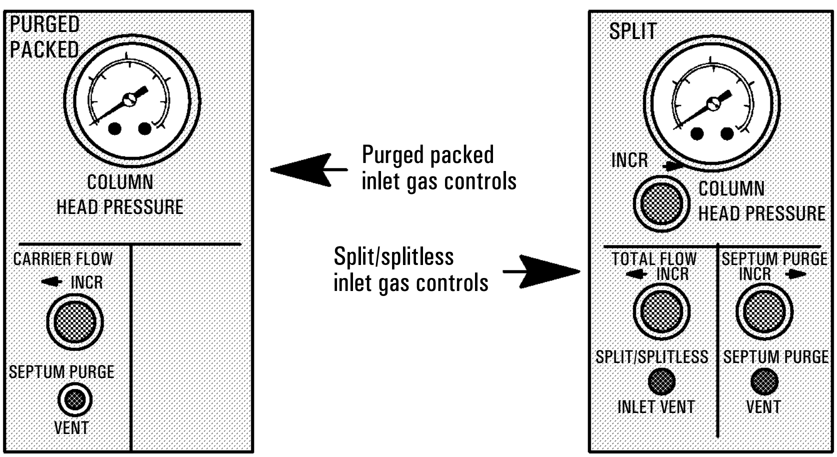


Figure 11 NonEPC inlet gas controls

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).

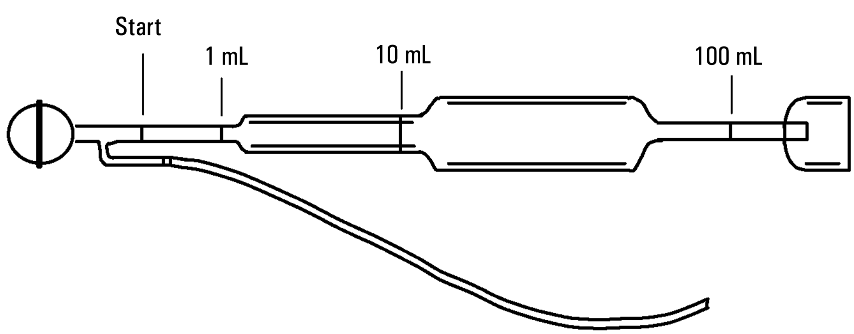
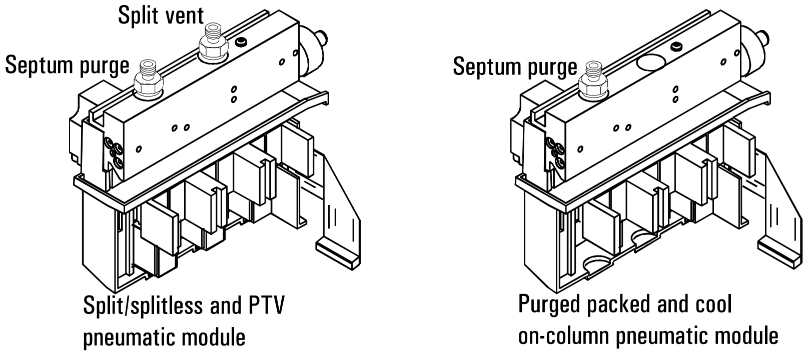


Figure 12 A three-volume bubble meter

Where to measure flows

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



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:

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

where:

Flow rate at NTP	is the flow rate in mL/min corrected to Normal Temperature (25°) and Pressure (1 atmosphere)
Flow $rate_{local}$	is the flow rate in mL/min measured by the bubble meter
$Temperature_{local}$	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).
$Pressure_{local}$	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.