Programmable Cool On-Column

HP 5890 Series II and HP 5890 Series II Plus

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Safety Symbols

This manual contains safety information that should be followed by the user to ensure safe operation.

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.

Important User Information for In Vitro Diagnostic Applications

This is a multipurpose product that may be used for qualitative or quantitative analyses in many applications. If used in conjunction with proven procedures (methodology) by qualified operator, one of these applications may be In Vitro Diagnostic Procedures.

Generalized instrument performance characteristics and instructions are included in this manual. Specific In Vitro Diagnostic procedures and methodology remain the choice and the responsibility of the user, and are not included.

Sound Emission Certification for Federal Republic of Germany

If Test and Measurement Equipment is operated with unscreened cables and/or used for measurements in open set-ups, users have to assure that under these operating conditions the Radio Interference Limits are still met at the border of their premises.

The following information is provided to comply with the requirements of the German Sound Emission Directive dated January 18, 1991

Sound pressure Lp < 70db(A)

During normal operation

At the operator position

According to ISO 7779 (Type Test)

When operating the HP 5890 Series II with cryo valve option, the sound pressure ≈ 78 db(A) during cryo valve operation for short burst pulses.

Schallemission

Werden Meß- und Testgeräte mit ungeschirmten Kabeln und/oder in offenen Meßaufbauten verwendet, so ist vom Betreiber sicherzustellen, daß die Funk-Entströbedingungen unter Betriebsbedingungen an seiner Grundstücksgrenze eingehalten werden.

Diese Information steht im Zusammenhang mit den Anforderungen der Maschinenlärminformation sverordnung vom 18 Januar 1991.

Schalldruckpegel LP < 70 dB(A)

Am Arbeitsplatz

Normaler Betrieb

Nach DIN 45635 T. 19 (Typprüfung)

Bei Betrieb des HP 5890 Serie II mit Cryo Ventil Option treten beim Oeffnen des Ventils impulsfoermig Schalldrucke Lp bis ca. 78 dB(A) auf.

Little Falls Site
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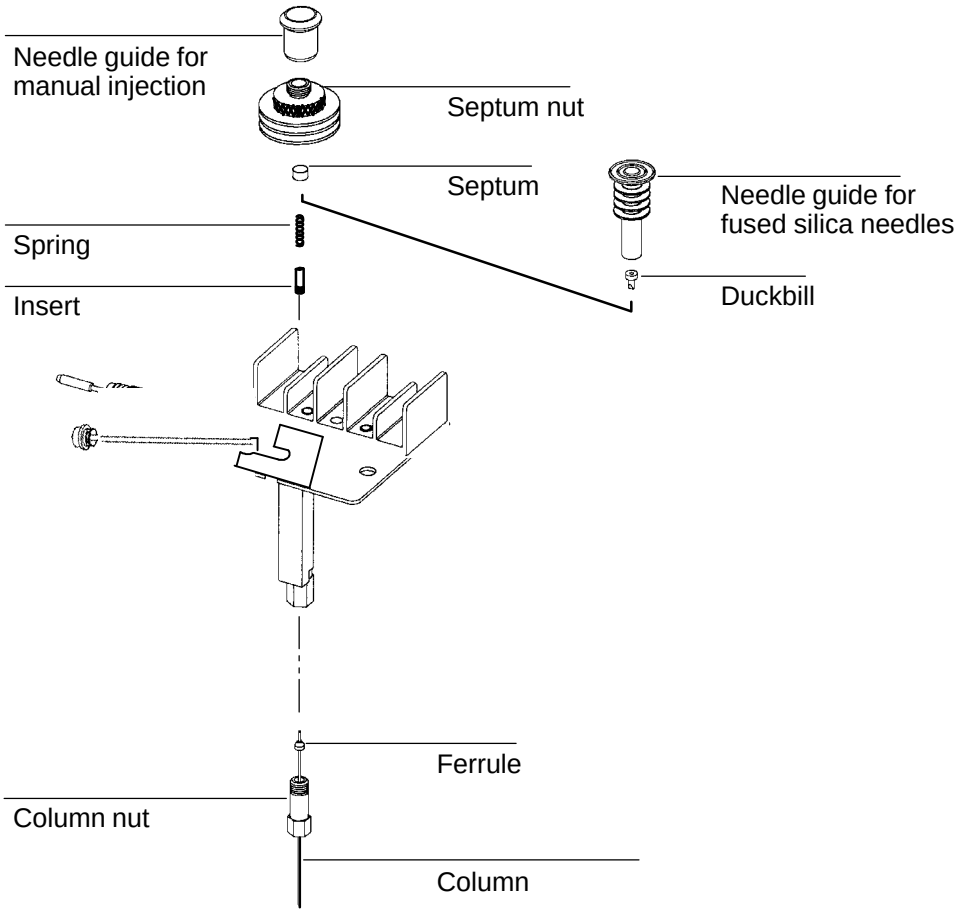
What is the Cool On-Column Inlet?

What is the Cool OnColumn Inlet?

The cool on-column inlet allows the introduction of small volumes of liquid sample into the column. Manual and automatic injection can be used to introduce sample into columns with internal diameters (id) of 250 μm, 320 μm, 530 μm, and larger.



Cool On-Column Inlet

Cool on-column injection is a technique of introducing a sample as a *liquid* directly onto a GC column. This lack of prior vaporization offers the following advantages:

- **Eliminates sample discrimination.** This is a main source of error in the quantitative analysis of samples covering a wide range of molecular weights. Inlet-related discrimination does not occur because the liquid is introduced directly onto the cool column.
- **Eliminates sample alteration.** Thermally labile components are not exposed to thermal stress because they begin the chromatographic process at relatively low temperatures. This also reduces decomposition and rearrangement reactions.
- **Provides high analytical precision.** If properly performed, on-column injection provides extremely accurate and precise results.

Cool on-column injection has extended the range of capillary gas chromatography to include many classes of compounds that until now were difficult, if not impossible, to analyze.

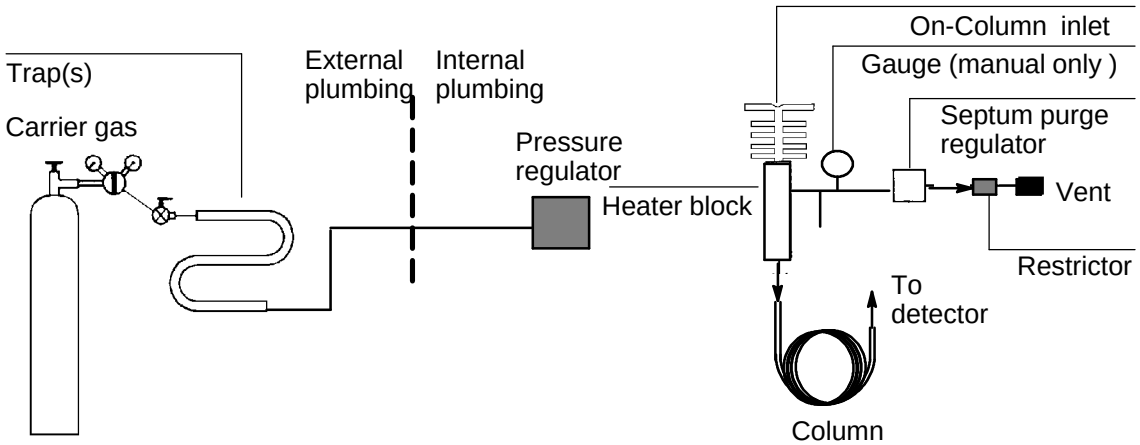
Compared with vaporizing inlets, the cool on-column inlet has some special requirements:

- **Requires relatively clean samples,** because the sample is introduced directly onto the column. Less volatile material can collect at the head of the column resulting in loss of separation efficiency, and can introduce adsorptive sites in the column inlet. When dirty samples are unavoidable, a retention gap can be helpful.
- **Real samples are often too concentrated** for on-column injection and must be diluted. As simple as this may seem, this often creates undesired peaks. Solvent impurities of milligrams per liter can disturb the sample profile. Subtraction of blank runs of the solvent used for dilution may be helpful, as may the use of automatic samplers with submicroliter capabilities.
- **Peak splitting or peak distortion** can occur due to differing polarities of solvent, stationary phase, and solutes. A retention gap can reduce these negative effects.

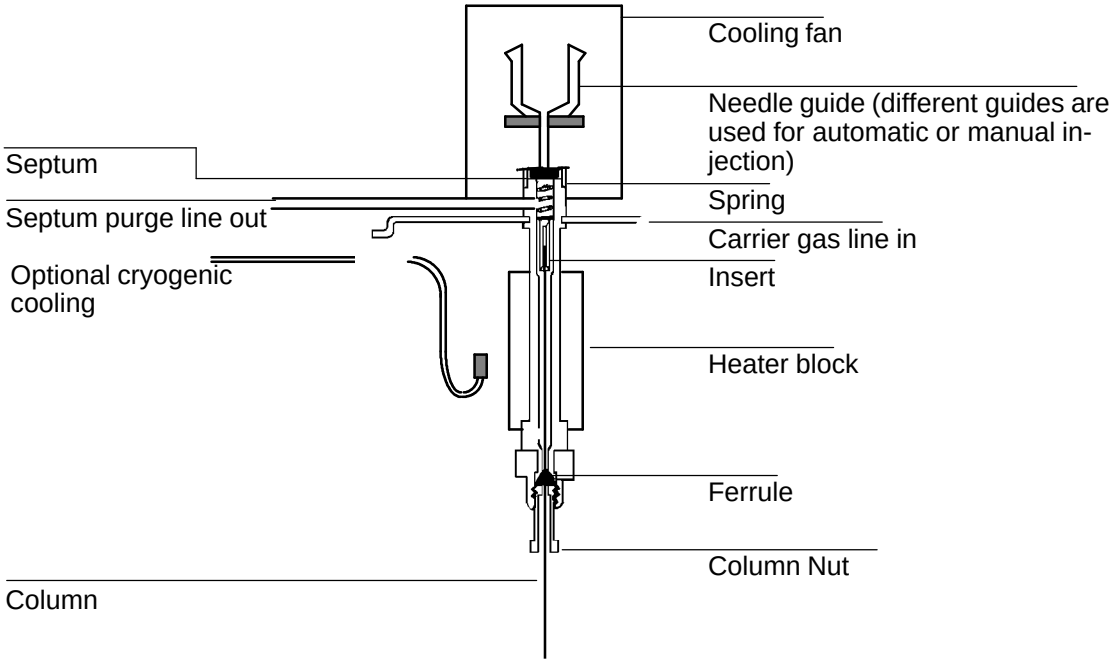
Features of the HP cool oncolumn inlet

The HP cool on-column inlet gives you the ability to inject samples directly onto a cool capillary column. Other features include:

- **Temperature programming.** Inlet temperature can be set to follow the oven program or a separate program with rates up to 100°C/min. A fan shortens the cool-down time between runs.
- **Pressure programming.** The electronic pressure programming option provides very accurate and precise control of column head pressure, resulting in retention time reproducibility of better than 0.02% RSD when there are no column effects. The inlet pressure can be constant, programmed, or set to maintain a desired column flow rate. Mass flow control can be maintained even at vacuum column outlet pressures. A safety shutdown feature stops the run if the column breaks or pressure otherwise falls.
- **Liquid CO_2 and N_2 cooling.** While an inlet cooling fan is standard, optional cryogenic cooling of the inlet and oven can shorten the cycle time between runs.
- **Flexibility.** Accommodates manual and automated injections with a conventional septa or septumless device onto 530 μ, 320 μ, and 250 μ columns or precolumns, and manual injections onto 200 μ columns.



Cool On-Column System Block Diagram



Cool On-Column Inlet Cross Section

For a complete list of the consumables needed to operate the cool on-column inlet, see Hewlett-Packard's analytical supplies catalog.

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Using the Correct Insert, Needle Guide, and Septum

Using the Correct Insert, Needle Guide, and Septum

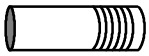
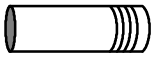
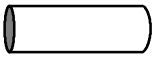
The consumables used with the cool on-column inlet are different for manual and automatic injection. For both injection techniques, the on-column inlet uses a small metal insert and a spring inside the inlet body to ensure that the syringe needle is guided smoothly into the column. The insert must be selected and installed before you install the column.

The correct insert and needle guide need to be installed before the column is installed. This chapter lists the procedures for selecting the correct insert

See the *Installing columns* section of this chapter for procedures for installing the column.

Selecting the correct insert

The small metal insert is installed in the inlet of the HP 5890 Series II GC to guide the syringe needle smoothly into the column. The insert must correspond to the size of the column and syringe needle you will use. Inserts are identified by the numbers of rings on them as shown in the table below. All the inserts can be used with automatic and manual injection.

Column Size and Type	Insert	Number of Identification Rings
250 μm Silica		6
320 μm Silica		5
530 μm Al coated		4
530 μm Polyimide coated		0

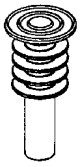
After you select the correct insert, use the instructions in the *Installing columns* section of this chapter to check the needle-to-column size before installing the column.

Changing the needle guide and septum

Two needle guides are available for the on-column inlet, depending on whether you are using fused silica or stainless steel syringe needles. The former guide requires a duckbill-type septum, while the latter uses a small, disk-type septum.

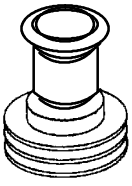
To automatically inject into 250 μm and 320 μm columns, use a 5 mm through-hole septum.

Fused silica needle guide



Duckbill septum

Stainless steel needle guide and finned septa retainer



Disk septum

5-mm through hole septum

Note: The stainless steel top needle guide is only used for manual injection or automatic injection with the HP 7673A Automatic Sampler. It can not be used with the HP 7673B Automatic Sampler.

How to change the needle guide and septum:

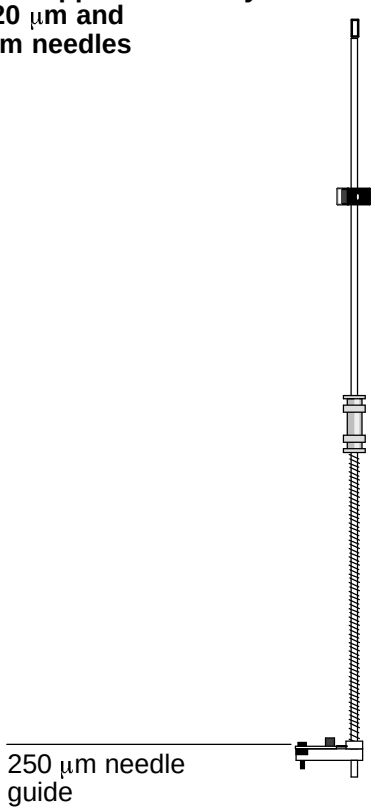
1. Turn the oven OFF and set inlet pressure to 0.
2. Unscrew and remove the septa retainer.
3. Find the old septum. It is either inside the needle guide, or at the top of the inlet base. If the septum is a disk septum, discard it. If it is a duckbill septum, you can reuse it if it has not been leaking.
4. Find the small coil spring either in the top of the inlet base, or on the bill" of a duckbill septum. Place the spring at the top of the inlet base, if not already there. Do not lose or damage the spring because it is required to keep the insert in position.
5. Replace the old septum with a new one appropriate for the needle guide you are using.

   Place the septum on top of the coil spring in the top of the inlet base. For a duckbill septum, make sure the bill" points DOWN, into the inlet base, and is inside the coil spring.
6. Install the new needle guide, tightening it finger-tight.

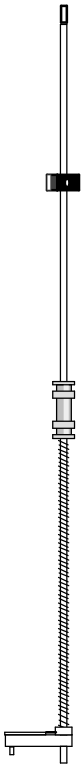
Selecting the correct needle support assembly

The following needle support assemblies are used for automatic injection into the on-column inlet.

Needle support assembly for 320 µm and 250 µm needles



Needle support assembly for large needles



Using retention gaps and other precolumns

Precolumns are columns connected to the front of the analytical column. They are commonly used to protect the analytical column from contamination.

A retention gap is a deactivated, uncoated (or thinly coated) precolumn. It is used to increase sample resolution and decrease peak splitting. Retention gaps have the effect of re-forming broad injection bands at the head of the column.

Retention gaps work well because when you first inject a sample, it exists as both gas vapor and microdroplets. Without a retention gap, the gas vapor begins partitioning immediately at the stationary phase. The microdroplets, however, are carried further into the column by carrier gas and cause loss of resolution and peak splitting. The addition of a retention gap in front of the column prevents this premature partitioning until all of the microdroplets are vaporized.

In general, the length of the retention gap required and type of deactivation depend on injected volume and solvent polarity. A working rule of thumb is to use between 0.3 and 1 m of retention gap per ml injected. For a 3 ml sample, use a retention gap that is between 1 and 3 m. The retention gap should be wetted by the solvent, which means it should be deactivated with material of similar polarity. Fused silica tubing is commercially available in a range of diameters and deactivations for this purpose.

Pressfit connectors

Press-fit connectors are easy-to-use, general-purpose connectors for coupling capillary columns of the same or different diameters.

Advantages: Press-fit connectors are inexpensive, have low dead volume, fit most fused silica columns, have low mass (no thermal lag), and are transparent. In most cases, simply pressing the column ends into the connector is the only installation task; heat from the oven completes the seal.

Disadvantages: Because press-fit connectors are made of glass, these connectors may be too reactive for some compounds. In addition, they may need to be heated separately from the sample for a reliable seal, and they expose a small amount of polyimide to the sample. If simply pressing the column ends into the connector does not give a good seal, try the following procedure:

1. Cut clean, square ends on the columns and wipe them with methanol to remove fingerprints and dust.
2. Grasp one end of the connector with a folded tissue to avoid burned fingers. Heat the other end with a hot-air gun (clamped in a ring stand so hands are free) for about 30 seconds. Remove the connector from the heat and immediately insert the column. Hold for about one minute while the fitting cools and shrinks around the column. Repeat with the other connection.
3. To finish forming the seal, place the press-fit connector in the gas chromatograph oven, set the temperature above 200°C, and run a low carrier gas pressure. You should now be able to see the polyimide seal.

Butt connectors

Butt connectors are popular for connecting precolumns to columns for high-temperature use. Different size ferrules are used, depending on the size of the columns. Because column ends are in contact with each other inside the ferrule during tightening, exposure to ferrule material is minimized.

Purged connectors

Purged connectors are commercially available for column connection. The most complex of connector types, they purge the connection area and thus minimize contamination.

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Installing Columns

Installing Columns

The correct insert and needle guide need to be installed before the column is installed. This section lists the procedures for selecting the correct insert, checking the column size, installing and aligning the inlet septum nut, installing the needle into the syringe barrel, and checking the column for installation problems.

The table below shows your choices of needles and the ability to automate depending on the column size.

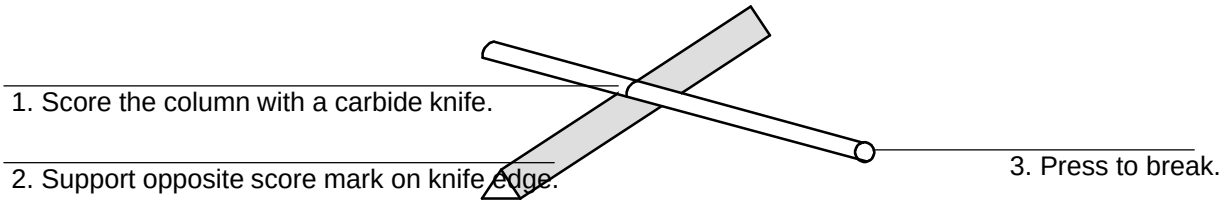
Column id	Can I do automatic injection?	Can I do manual injection with a fused silica needle?	Can I do manual injection with a stainless steel 26-gauge needle?
0.53 mm[1]	Yes	Yes	Yes
0.32 mm	Yes	Yes	Yes, you use 26-32 gauge (HP 5181-1266).
0.25 mm	Yes	Yes	Yes, you use 26-32 gauge (HP 5181-7442).
0.2 mm	Yes[2]	Yes	Yes[2]

1. This includes aluminum-coated columns.
2. If you use a 0.25 or 0.32 mm precolumn.

Preparing a fused silica capillary column

This method gives a square cut for the column. If you are using precolumns or retention gaps, see *Using the correct insert needle guide, and septum* earlier in this chapter to select the right one, and connect it to the analytical column before you continue.

1. Cut off the column end with a square cut according to the illustration below.



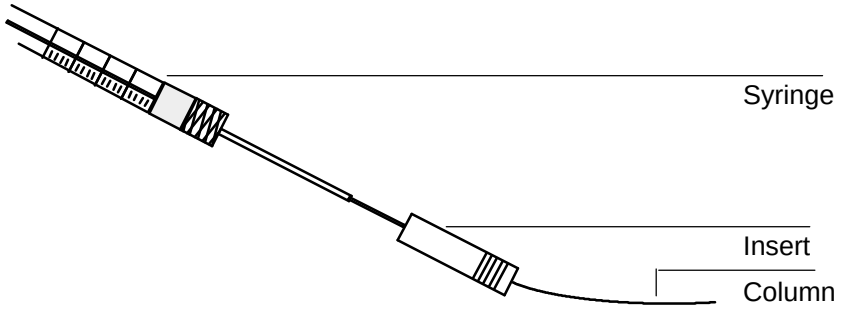
2. Wipe the column end with methanol to remove fingerprints and dust.

WARNING Flying glass particles can cause eye injuries. Always wear safety glasses when cutting fused silica columns.

Checking the needletocolumn size

After selecting an insert and before installing the column, you need to check the needle-to-column size because some manufacturers provide columns with ids that are too small. You may bend your 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 by the number of rings on it.
2. Insert the column into one end of the insert as shown below.

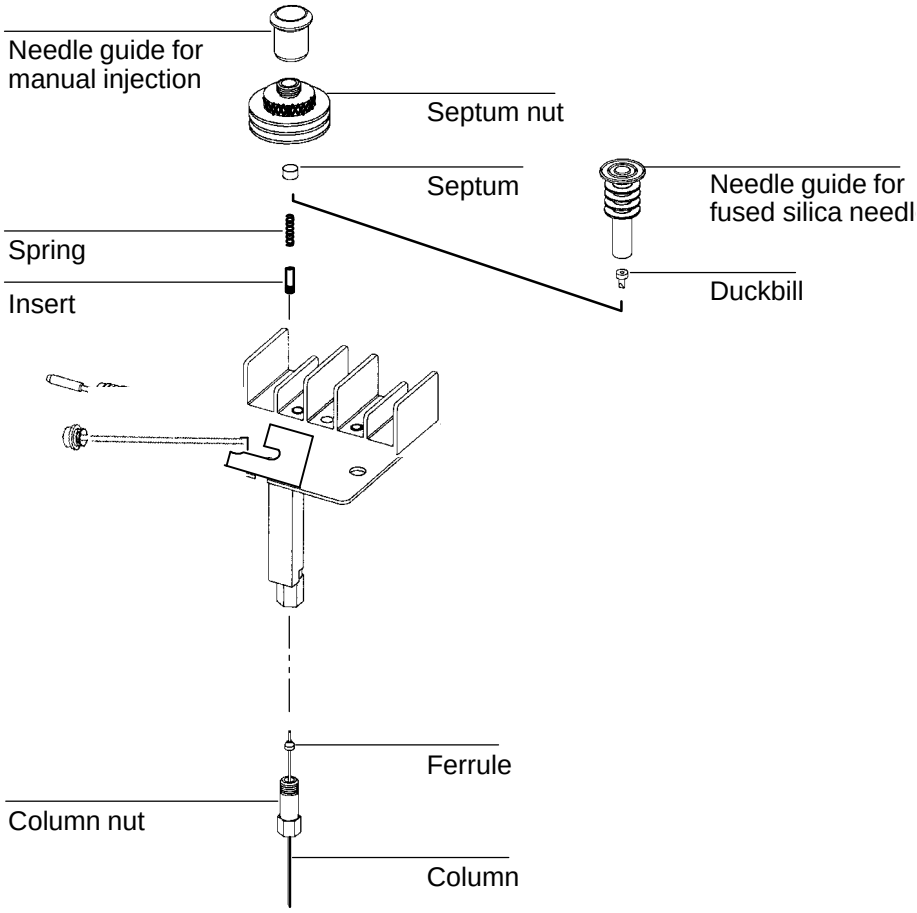


3. Insert the syringe needle through the other end of the insert and into the column. If the needle cannot pass easily into the column, reverse the insert to try the needle and column in the other end.
4. Notice into which end of the insert the column fits because that will be the end that goes into the inlet first when you install the insert.

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.

Installing the insert

1. Turn off the GC oven and set the inlet pressure to 0.
2. Remove the column, column nut, and ferrule.
3. On top of the oven, unscrew and remove the inlet septum retainer.
4. Replace the existing septum with a new one, and set the inlet septum retainer aside. If the septum remains in the needle guide, do not remove it unless you want to change it.



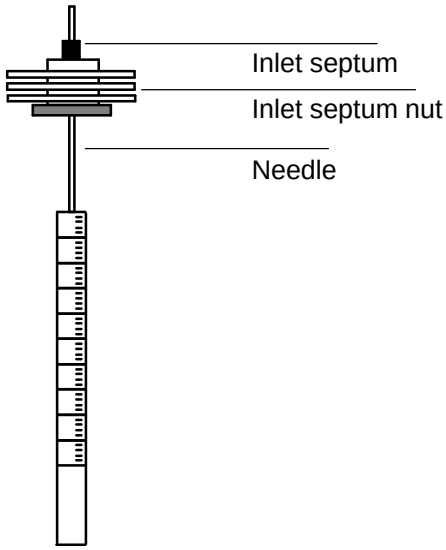
Cool On-Column Inlet

5. Remove the spring from the inlet, 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 pushing it out from below with a piece of column. Store the insert for possible later use.
7. Drop the new insert straight into the inlet from the top. The end of the insert in which you inserted the needle should be facing up; the end of the insert in which you inserted the column should be facing down (see the previous section on checking the column size). Use the identification rings to help you remember which way to install the insert.
8. Replace the spring on top of the insert.
9. Follow the instruction in the next section to install the inlet septum nut and reassemble the inlet.

Aligning the inlet septum nut

The inlet septum nut needs to be properly aligned to avoid bending the syringe needles during injection.

1. To ensure proper alignment of the through-hole septa, thread the septum and the septum nut onto a 26-gauge or larger needle or a piece of column or wire. The septum should rest in the cavity in the nut as shown below. For 250 μm and 320 μm on-column injections, the septum nut requires one of the 5 mm through-hole septa included in the accessory kit.
2. Turn the needle over, and insert the wire, inlet septum nut, and septa into the inlet.



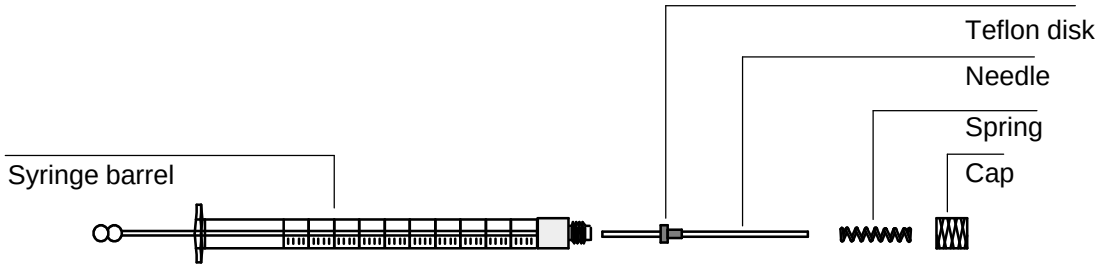
This end is inserted into the inlet.

3. Tighten the septum nut fairly tightly so that you get a good seal.
4. Remove the needle from the inlet.

Installing the needle into the syringe barrel for automatic injection

The stainless steel needles used for 250 μm and 320 μm injections must be inserted into a glass syringe barrel before they can be used in the HP 7673 Automatic Sampler.

1. Unscrew the syringe barrel cap, and remove the spring.
2. Select the correct size needle for the column you plan to use, and make sure the needle has a Teflon disk. If the syringe barrel does not have the Teflon disk, use the instructions in the syringe box to wrap the needle yourself.



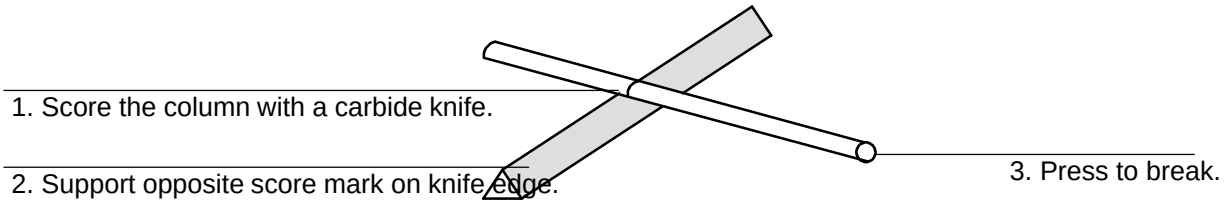
3. Slide the spring and the cap down over the needle.
4. Insert the needle into the syringe barrel.
5. Screw the cap back on the syringe barrel.

Caution Do not operate the injector without a syringe in place because the syringe latch may interfere with the motor if it is allowed to swing freely.

Installing a fused silica capillary column for automatic injection

Column connections will be more successful if you prepare the columns properly. If you have not prepared your column, use the instructions below to do so now.

1. Install a column nut and ferrule on the end of the column.
2. Cut off the column end with a square cut according to the illustration below.

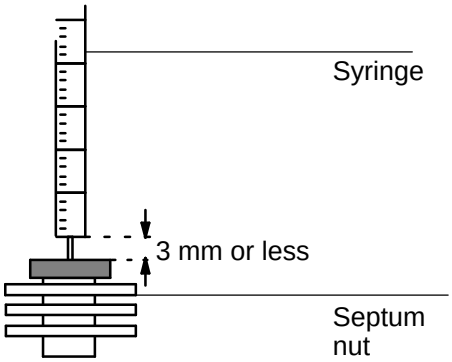


3. Wipe the column end with methanol to remove fingerprints and dust.
4. Insert the column into the inlet base until it cannot go further.

WARNING Flying glass particles can cause eye injuries. Always wear safety glasses when cutting fused silica columns.

5. Finger tighten the column nut, making sure the column remains against the insert.
6. Use a wrench to tighten the column nut an additional 1/4-turn.
7. Verify the column installation by manually pushing your syringe into the inlet. There should be a gap of 3 mm or less between the septum nut and the syringe barrel.

If there is more than 3 mm between the syringe barrel and septum nut, your needle is not reaching the column, and you will not be able to automate injection onto 250 μm and 320 μm columns.



Checking for column installation problems

Using the right ferrules

Ferrules that are the wrong size or are improperly prepared cause leaks and contamination. Here are some hints to avoid problems:

- **Graphite-containing ferrules** should be baked at 250-300 °C for 30 minutes before use because graphite is a good absorbent for organic compounds present in the atmosphere. Leave a petri dish of assorted ferrules in the GC oven to ensure a clean supply.

  The ferrule should slide easily on the column, but not fall off under its own weight. In this case, no more than 1/4-turn from finger tight is required to make a good seal.

  If the column od is significantly smaller than the ferrule inside diameter, the column nut will have to be tightened enough to compress the ferrule around the column. This is usually not a problem with easily deformed graphite ferrules. Harder ferrules may require so much torque that the inlet fitting may be bent, or the nut may be broken. In addition, the ferrule may split and yield a leak. With hard ferrules, it is better to start with an undersize hole and drill it to fit the column.

- **Vespel ferrules** can be more leaktight than graphite, but have a lower temperature limit. They should be retightened after a few temperature cycles to ensure a good seal.

  Be sure to use the correct ferrule for the size column you are using.

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Setting Inlet Pressure

Setting Inlet Pressure

Your cool on-column inlet is equipped with either manual pressure control or electronic pressure control. Read *Selecting the best inlet pressure* in this section, and then go to the instructions for either manual or electronic pressure control, depending on your unit.

Selecting the best inlet pressure

This table is a starting point for selecting pressure, based on column length and bore.

		Length (m)		
		12	**25**	**50**
id (mm)	**0.20**	135 (19.6)	223 (32.4)	347 (50.3)
	0.32	45 (6.5)	82 (11.9)	137 (19.9)
	0.53	11 (1.7)	23 (3.3)	42 (6.1)

Assumptions:
Carrier gas: He
Temperature: 100°C
Optimal linear velocity: 30-60 cm/sec.

Pressure at Optimal Linear Velocity in kPa (psi) Based on Column id and Length

When using the 5 m x 0.53 mm id checkout column, suggested pressure is 15 kPa (2.2 psi) with helium gas flow of 20 ml/min.

Choosing pressure for a desired linear velocity

Linear velocity through the column is measured by injecting a sample containing an unretained component (typically CH_4).

The observed retention time for the unretained component is compared to an expected retention time (t_r) calculated from the desired linear flow velocity (μ) and the length of the column:

$$t_{r\ expected}\ \text{(in min)} = 1.67 \times \frac{\text{Column length (m)}}{\text{Linear flow velocity (cm/sec)}}$$

With repeated injection of the unretained component, adjust the pressure as necessary to obtain the expected retention time for the desired linear velocity.

To verify flow rate through the column:

1. Turn off detector gases.
2. Measure flow using a bubble flow meter connected at the detector exhaust vent.

Checking vent flow rate

The septum purge vent is not adjustable, but you should check the flow. Do not cap off the flow from the purge vent.

Carrier Gas Type	Approximate Flow
H_2	12 ± 3 ml/min
He	12 ± 3 ml/min
N_2	12 ± 3 ml/min
Ar - Me	12 ± 3 ml/min

WARNING Hydrogen gas is flammable. If you use hydrogen carrier gas, either vent the purge flow appropriately or add a needle valve restrictor at the vent fitting and adjust the flow down.

Safety shutdown

Programmable cool on-column inlets that are equipped with electronic pressure control have a safety shutdown feature to prevent gas leaks from creating a safety hazard. If the system cannot reach a pressure setpoint, the system beeps. After about two minutes, the beeping stops and the following message appears on the display:

The system will shut down by turning off all electronic pressure and heated zones and locking the keyboard.

See the *Maintanence and troubleshooting* section in this chapter for more information about safety shutdown.

Note: These safety shutdown procedures apply to EPC boards with a mainboard ROM part number HP 05890-80310 or higher.

Setting inlet pressure using electronic pressure control

If your GC is equipped with electronic pressure control, you can set constant pressure or create a pressure program with multiple ramps.

Before you start

1. Be sure carrier source pressure is at least 138 kPa (20 psi) greater than the maximum desired inlet pressure.
2. Select the pressure units you would like to use.
   To change the units, press gold 1 ENTER, and then select
   1 = psi,
   2 = bar, or
   3 = kPa.
3. From the keyboard, set the pressure.

Set the oven and heated zone temperatures. Be sure the detector is turned on, and its output signal is assigned to the correct channel.

Zeroing pressure

An inlet with electronic pressure control is zeroed when it is shipped. However, you should check this periodically. To zero the pressure, press:

1. gold INJ B PRES 0 . 0 ENTER *Set inlet B pressure to 0.0.*

   Allow enough time for the column to completely depressurize. Remove the septa retainer or disconnect the source pressure to depressurze the column more quickly.

2. To zero channel B, press gold 3 ENTER *value* ENTER

   where *value* is the zero offset value shown on the GC display labelled actual".

3. gold INJ B PRES 0 . 0 ENTER *Set inlet B pressure to 0.0.*

   Allow enough time for the column to completely depressurize. Remove the septa retainer or disconnect the source pressure to depressurze the column more quickly.

4. To zero channel A, set inlet A pressure to 0.0, then press

   gold 2 ENTER *value* ENTER

   where *value* is the zero offset value shown on the GC display labelled actual".

Setting constant pressure

Follow the example below to set Injector B pressure to 10 psi.

gold INJ B PRES 1 0 ENTER *Set inlet B pressure to 10 psi.*

	ACTUAL	SETPOINT
EPP B	**10.0**	**10.0**

The GC display looks like this.

INIT TIME 6 5 0 ENTER *Set initial time to 650 (max).*

Note: If the initial time is less than the total run time, the inlet B pressure will change because it is being controlled by the constant flow mode. Turn off the flow control option to avoid having the pressure change during the run. To do this, press the following keys until the display looks like the one below:

gold FLOW PARAM OFF ENTER
ACTUAL SETPOINT

EPP B CONST FLOW OFF

The GC display looks like this.

Setting pressure programs (1 Ramp)

This example shows how to start with Injector B pressure at 10 psi for 1 minute, then ramp at 5 psi/min to 20 psi, and remain there for 2 minutes.

gold INJ B PRES INIT VALUE 1 0 ENTER	*Set Injector B initial pressure at 10 psi.*
INIT TIME 1 ENTER	*Set initial time at 1 minute.*
RATE 5 ENTER	*Set ramp at 5 psi/min.*
FINAL VALUE 2 0 ENTER	*Set final pressure at 20 psi.*
FINAL TIME 2 ENTER	*Set final time at 2 minutes.*

Note: The oven program determines the run time of the analysis. If the inlet pressure program is shorter than the oven temperature program, the inlet pressure will go into the constant flow mode for the remainder of the run. To prevent this, set the pressure program longer than the oven temperature program.

Setting pressure programs (2 and 3 Ramps)

To add a second and third ramp to the above pressure program, press the following keys after you enter the previous example:

RATE A 2 ENTER	*Set the second ramp at 2 psi/min.*
FINAL VALUE A 2 6 ENTER	*Set the second final pressure at 26 psi.*
FINAL TIME A 2 ENTER	*Set the second final time at 2 minutes.*
RATE B 4 ENTER	*Set the third ramp at 4 psi/min.*
FINAL VALUE B 3 0 ENTER	*Set the third final pressure at 30 psi.*
FINAL TIME B 5 ENTER	*Set the third final time at 5 minutes.*

Checking the pressure program

Display the pressure program by pressing any pressure program key, followed by ENTER. Successively press ENTER to scroll through the program.

Note: The oven program determines the run time of the analysis. If the inlet pressure program is shorter than the oven temperature program, the inlet pressure will go into the constant flow mode for the remainder of the run. To prevent a pressure program from going into constant flow mode, be sure to set the pressure program longer than the oven temperature program.

Using the constant mass flow modes

If your system is equipped with electronic pressure control, you can program the inlet to maintain constant mass flow. To use the constant mass flow modes:

1. Select the gas type.

gold FLOW PARAM FLOW PARAM — *Scroll to select gas type.*

	ACTUAL	SETPOINT
EPP B	**He**	**[1]**

The GC display now looks like this. Press
1= Helium, 2 = Nitrogen, 3 = Hydrogen, 4= Argon/Methane.

2. Press FLOW PARAM again until you see the constant flow display.

	ACTUAL	SETPOINT
EPP B	**CONST FLOW OFF**	

3. Select the option you wish to use.

   OFF = constant flow mode off.

   ON = constant flow mode on. You set pressure at oven initial temperature after turning on the constant flow feature.

Turning off constant flow

When you select OFF, you turn off the constant flow mode. You must select OFF if you wish to create independent pressure programs.

Turning on constant flow—you set initial pressure, and the GC maintains initial flow

When you select ON, you turn on the constant flow mode. You can set an initial pressure at oven initial temperature as follows:

gold INJ B PRES 1 0 ENTER — *Set starting pressure to give a constant flow.*

The GC will then maintain the initial flow throughout the run by automatically adjusting pressure.

Note: Make sure the oven is equilibrated to initial temperature when the initial pressure is set; an incorrect flow will result if initial pressure is set before initial oven temperature is reached.

Constant flow hint

You can check the flow through the column by turning off the detector gases and measuring the flow using a bubble flow meter and the GC stopwatch feature. Check the Operating Manual for more details.

Setting mass flow rate

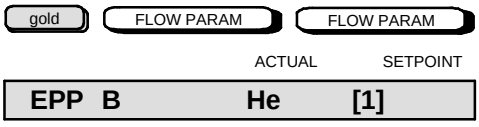
If your system is equipped with electronic pressure control, you can set mass flow rate. This flow control capability is accomplished through the electronic pressure control function.

For example, when a constant pressure is set, mass flow is displayed (monitoring mass flow). Entering a new mass flow value will automatically set a new constant pressure to obtain the flow value entered.

Note: Before setting mass flow, the correct column parameters (column length, column diameter, and gas type) must be entered.

Follow the example below to set Injector B flow to 10 ml/min.

1. Select the gas type.

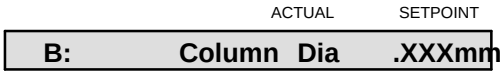


Scroll to select gas type.

The GC display now looks like this. Press
1 = Helium, 2 = Nitrogen, 3 = Hydrogen,
4 = Argon/Methane.

2. Enter the column diameter.

   Press FLOW PARAM again until you see the column diameter (id) display.



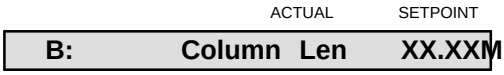
The GC display looks like this.

   The column diameter must be entered in microns (i.e., 200 μm, 530 μm).

   5 3 0 ENTER *Sets the column diameter to 0.530 mm.*

3. Enter the column length.

   Press FLOW PARAM again until you see the column length display.



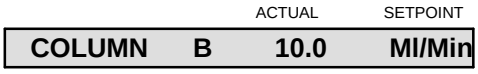
The GC display looks like this.

   Column length must be entered in meters. If exact column length is unknown, refer to *Determining corrected column length*.

   2 5 ENTER *Sets the column length to 25 meters.*

4. Set the column B desired flow rate.

   Press CLEAR FLOW until you see the mass flow control display.



The GC display looks like this.

   1 0 ENTER *Sets inlet B flow to 10 ml/min.*

   Note: Inlet B pressure will change to a value needed to produce 10 ml/min.

Determining the corrected column length

When you want to set or display mass column flow, you must determine a corrected length and diameter for an equivalent open tubular column.

1. Press gold FLOW PARAM FLOW PARAM until you see the column diameter display.

   Set column diameter to its nominal inner diameter value (100 μm, 200 μm, 530 μm, etc.). For a packed column, enter 530 μm.

2. Press FLOW PARAM until you see the column length display.

   Set column length to its nominal length in meters.

3. Select the gas type.

   Press gold FLOW PARAM FLOW PARAM *Scroll to select gas type.*

	ACTUAL	SETPOINT
EPP B	**He**	**[1]**

   The GC display now looks like this. Press 1= Helium, 2 = Nitrogen, 3 = Hydrogen, 4 = Argon/Methane.

4. Press CLEAR FLOW FLOW until the display reads:

		ACTUAL	SETPOINT
COLUMN	**B**	**97.5**	**Cm/Sec**

   This is the computed average linear velocity ($\bar{u}$) for the estimated length column (10 m) at the given temperature.

5. Inject an unretained component and determine its retention time in minutes. This is t_o Actual.

6. Calculate corrected column length as:

$$L_{corrected} = \left(\frac{t_o \text{ Actual} \times \bar{u}}{1.67} \right) \text{ where:}$$

$L_{corrected}$ = corrected column length in meters
t_o Actual = retention time of unretained component in minutes
$\bar{u}$ = average linear velocity in cm/sec

Enter the value calculated from the above formula as the column length in step 3 of *Setting mass flow*.

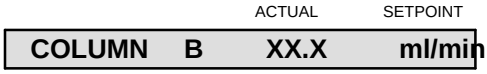
Setting flow programs

Flow programs can be set indirectly through pressure programming once gas type, column diameter, and column length have been entered.

Note: Column length must be entered in meters. If exact column length is unknown, or if a packed column is in use, refer to *Determining corrected column length*. This example shows how to obtain pressure values necessary to create a pressure program to set column B flow at 4 ml/min, then end with 7 ml/min.

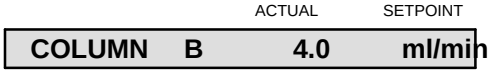
This example shows how to obtain pressure values necessary to create a pressure program to set column B flow at 4 ml/min, then end with 7 ml/min.

1. Press CLEAR FLOW FLOW until the display reads:



The display looks like this.

2. Press 4 ENTER until the display reads:

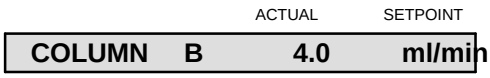


The display looks like this.

3. Press gold INJ B PRES until the display reads:

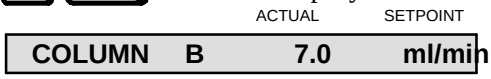
The GC display looks like this.
This will be injector B pressure initial value.

4. Press FLOW FLOW until the display reads:



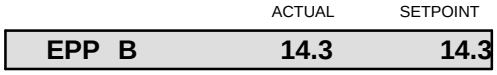
The display looks like this.

5. Press 7 ENTER until the display reads:



The display looks like this.

6. Press gold INJ B PRES until the display reads:



The GC display looks like this.
This will be injector B pressure final value.

Use the pressure values obtained from this procedure when you set a pressure program. Initial time, ramp rate, and final time are entered as part of setting the pressure program (see *Setting pressure programs*).

Setting average linear velocity

If your system is equipped with electronic pressure control, you can set average linear velocity. Like mass flow control, the average linear velocity capability is accomplished through the electronic pressure control function.

For example, when a constant pressure is set, average linear velocity is displayed (while you are monitoring average linear velocity). Entering a new average linear velocity value will automatically set a new constant pressure (and flow rate) to obtain the velocity value entered.

Note: Before setting average linear velocity, the correct column parameters (column length, column diameter, and gas type) must be entered.

To set average linear velocity, perform steps 1 through 3 of *Setting mass flow*. Then follow the example below to set the average linear velocity to 100 cm/sec.

1. Set the column B desired average linear velocity.

   Press CLEAR FLOW FLOW until the display reads:

   ACTUAL SETPOINT

COLUMN B 97.5	**Cm/Sec**

   The GC display looks like this.

   Press 1 0 0 ENTER *Set inlet B linear velocity to 100 cm/sec.*

   ACTUAL SETPOINT

COLUMN B 100.0	**Cm/Sec**

   The GC display looks like this.

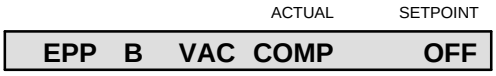
Note: The pressure and flow for Inlet B will change to a value needed to produce 100 cm/sec.

Using vacuum compensation mode

Vacuum compensation should be used anytime a mass spectrometer is used, to allow column outlet pressure to be at vacuum conditions. With vacuum compensation and constant flow on, a constant mass flow is delivered to the mass spectrometer throughout a temperature programmed run. This results in a constant mass spectrometer ionization current level and a 15 to 30% shorter run time.

1. Turn on vacuum compensation:

   Press gold FLOW PARAM FLOW PARAM until you see the column compensation display.



The GC display looks like this.

   To turn on column compensation, press ON.

   To turn off column compensation, press OFF.

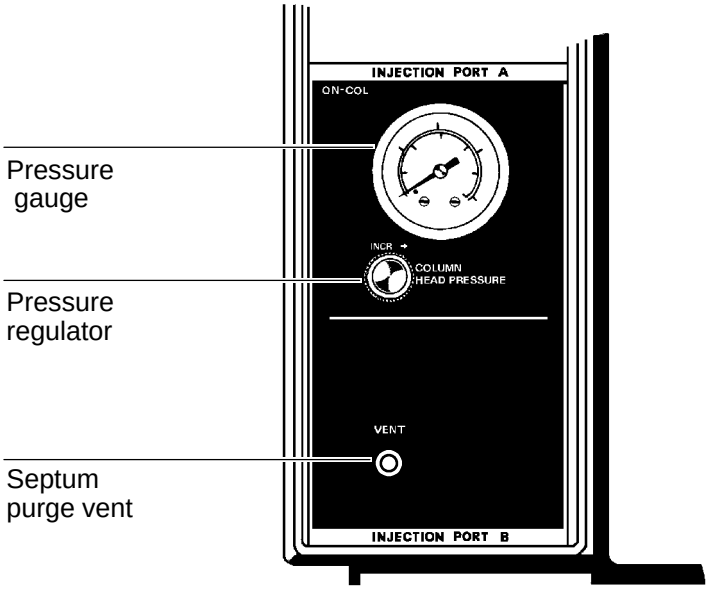
After you turn on the vacuum compensation feature, you enter the desired pressure setting.

Note: HP 5890 Series II GC instruments with electronic pressure control that were built before July 1, 1990 will display the message change EPC ROM. When this occurs, contact Hewlett-Packard to get an updated ROM.

Setting pressure using manual control

If your GC is equipped with manual pressure control, follow these instructions to set pressure.

1. Set oven and heated zone temperatures. Be sure the detector is turned on, and the output signal is assigned to the correct channel.



Flow Panel for Manual Pressure Control

2. Be sure the carrier source pressure is at least 138 kPa (20 psi) greater than the selected inlet pressure.
3. Select the inlet pressure using the pressure regulator.

Setting Inlet Temperature

Setting Inlet Temperature

Guidelines for selecting inlet temperature

1. Determine the oven temperature program that gives you good component separation. The starting inlet temperature is usually similar to the starting oven temperature, which is at or below the boiling point of the solvent.
2. Determine the total time of the run.
3. To save time between runs, set the inlet temperature program time shorter than the total time of the run. This way, the inlet will begin the cooling process earlier.
4. For best retention time reproducibility, use an oven equilibrium time of about 3 minutes (1 minute if cryogenic cooling is used).

Setting the inlet temperature to follow the oven temperature (oven track)

The oven track feature sets the inlet temperature 3°C higher than the oven at all times to optimize repeatability. Oven track is on when you receive your GC from the factory. To turn on oven track:

gold OVEN TRACK ON

Turn on the oven track option.

The GC display looks like this.

Creating an independent inlet temperature program

This example shows how to start with Injector B temperature at 60°C for 1 minute, then ramp at 20°C/min to 100°C, and remain there for 2.2 minutes.

INJ B TEMP INIT VALUE 6 0 ENTER	*Set initial inlet temperature.*
INIT TIME 1 ENTER	*Set initial time.*
RATE 2 0 ENTER	*Set initial rate.*
FINAL VALUE 1 0 0 ENTER	*Set final temperature.*
FINAL TIME 2 . 2 ENTER	*Set final time.*
INJ B TEMP INIT VALUE ENTER ENTER	*Scroll through inlet program.*

To optimize repeatability when creating inlet temperature programs, be sure that the inlet temperature is always at least 3° higher than the oven temperature. You do this by turning on the oven track mode.

If the message INJ B Oven Track On appears on the display, press OFF to disable the oven temperature tracking. You can now program the inlet temperature independently of the oven temperature.

Using cryogenic oven cooling

Oven temperature may be controlled below ambient when a cryogenic valve is present. Cryogenic control setpoints are described in the following table:

Cryogenic Control Setpoint	Description
CRYO ON	Enables subambient control of the oven.
CRYO OFF	Disables subambient cooling of the oven; the default state for the cryogenic valve is off.
CRYO BLAST ON	Enables very fast cooldown time after a run.
CRYO BLAST OFF	Disables very fast cooling of the oven.
AMBIENT	Sets optimal temperature control for efficient use of cryogenic fluid. The default temperature setting is 25 °C.
CRYO FAULT ON/OFF	A fault occurs when the oven does not reach set temperature after 17 minutes of continuous cryo operation. The oven turns off, and the message WARN:OVEN SHUT OFF is displayed. Turning off Cryo Fault will disable this feature.
CRYO TIMEOUT XXX MIN	A cryo timeout occurs when a run does not start within a specified time (10 to 120 minutes) after the oven equilibrates. Turning off Cryo Timeout will disable this feature. The default is ON for 30 minutes.

When the cryogenic valve is turned on, it operates automatically to obtain an oven temperature when there is demand for coolant to be supplied to the oven.

When cryogenic cooling is not needed, cryogenic valve operation must be turned off. If this is not done, proper oven temperature control may not be possible, particularly at temperatures near ambient.

The Cryo Blast feature can operate together with or independently of Cryo On/Off. Cryo Blast cools the oven faster after a run than it would under normal cryogenic operation. This allows the HP 5890 GC to be ready for the next run earlier than it would without Cryo Blast on. This feature is useful when maximum sample throughput is necessary.

Successively pressing the CRYO PARAM key scrolls through the cryogenic valve operation functions. Use the following key sequence to turn cryogenic operation or blast on and off:

gold → CRYO PARAM → ON → ON or OFF

To turn Cryo Blast operation on or off, use the following key sequence:

Press gold → CRYO PARAM → Scroll to Cryo Blast and press ON → ON or OFF

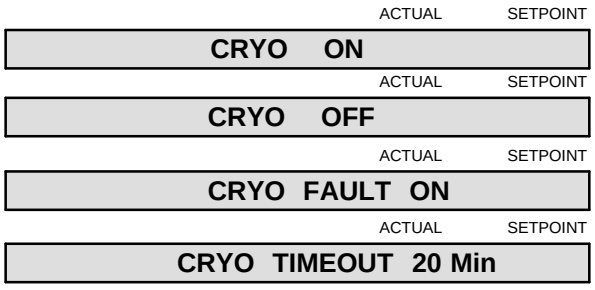
An example key sequence to change the ambient temperature setting to 23°C is:

gold → CRYO PARAM → Scroll to Ambient → 2 → 3 → ENTER

You can set the ambient temperature to allow fine tuning of cryogenic operation. The default setting is 25°C. It does not need to be changed for most applications. For more information about adjusting the ambient cryogenic setting, see the *HP 5890 Series II Reference Manual.*

The following figure shows the displays associated with disabling and enabling automatic cryogenic valve operation.

Automatic Cryogenic Valve Displays



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6

Making Injections

Making Injections

The HP 5890 Series II cool on-column inlet allows you to inject sample volumes onto columns that are 250 μm or larger using automatic and manual injection techniques.

Before the injection

Here is a checklist for injecting into a cool on-column inlet:

- The initial oven temperature should be 25°C below the boiling point of the solvent.
- The sample injection must be done quickly and smoothly to ensure that the sample is introduced in liquid form.
- The syringe needle must be withdrawn immediately after injection.
- The sample amount injected, both total volume and component concentration, is important; large volumes may cause band broadening, particularly for components with boiling points approximately 150°C greater than that of the solvent.
- The recommended sample injection volume is 0.5 μl or less for 0.20 mm columns. For injections with the HP 7673A automatic sampler that are less than 1 μl , use the nanoliter adapter kit.
- With automatic injection, make sure your syringe and inlet assembly are aligned correctly, or you may bend your needle.

Can I automate?

Yes, the special considerations for automatic injection are listed later in this chapter. The following table shows your choices of needles and automating ability based on the column used.

Column id	Can I do automatic injection?	Can I do manual injection with a fused silica needle?	Can I do manual injection with a stainless steel 26-gauge needle?
0.53 mm[1]	Yes	Yes	Yes
0.32 mm	Yes	Yes	Yes, you use 26-32 gauge (HP 5181-1266).
0.25 mm	Yes	Yes	Yes, you use 26-32 gauge (HP 5181-7442).
0.2 mm	Yes[2]	Yes	Yes[2]

1. This includes aluminum-coated columns.
2. If you use a 0.25 or 0.32 mm precolumn.

Manual injection technique with stainless steel needles

When you are injecting by hand using stainless steel needles, make sure the stainless steel needle guide and disk-type septum are installed. To perform the injection:

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 it if it is wet.

5. Guide the needle straight into the needle guide, pierce the septum, and insert the needle fully into the inlet.
6. Start the run, depress the syringe plunger *as quickly as possible*, and withdraw the needle from the inlet.

Note: These steps should be done smoothly, with minimal delay.

Manual injection technique with fused silica needles

Note: When injecting with fused silica needles, be sure the initial pressure is less than 30 psi. Higher pressures will make needle insertion difficult.

When fused silica needles are required for injection onto 320 µ and smaller diameter columns, follow these steps for injection:

1. Immerse the syringe needle in 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 the needle guide fully down to open the duckbill.

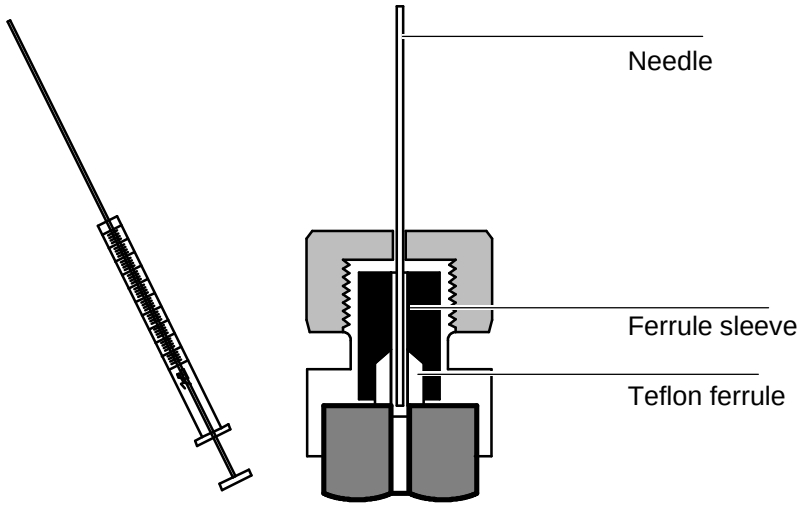
WARNING The inlet needle guide may be hot! Use a pencil to depress the needle guide.

5. Hold the needle guide down for about 3 seconds to depressurize the column. This prevents loss of sample due to backflow of carrier gas. When you do this with an electronic pressure controlled system, it will start to beep while the column is depressurised. The beeps will stop after the system repressurises.
6. While holding the needle guide down, guide the needle straight into the needle guide and fully insert it into the inlet.

   Hint: If the needle does not go in all the way, try rotating the syringe and slightly releasing pressure on the needle guide.

7. Once the needle is fully inserted, release the needle guide. Allow 1 to 2 seconds for backpressure on the duckbill to seal it around the inserted needle.
8. Start the run, depress the syringe plunger as quickly as possible, and withdraw the needle from the inlet.

Replacing the fused silica syringe needle



1. If you are cutting replacement needles directly from fused silica column material:
   a. Column material for making needles must have an od *smaller* than both the id of the on-column inlet (0.23 mm) and the id of the installed column.
   b. Column material must be washed free of active stationary phase.
   c. 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 ± 5 mm length to use as the syringe needle.

2. 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 may be blocked. You may need to replace the ferrule.

3. 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.

4. 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.

5. Use a suitable solvent to rinse the syringe and check for leaks or blocks.

   Leaks (inability to eliminate air bubbles) *may* be fixed by further tightening the retaining nut. Blocks (or serious leaks) require repeating this procedure.

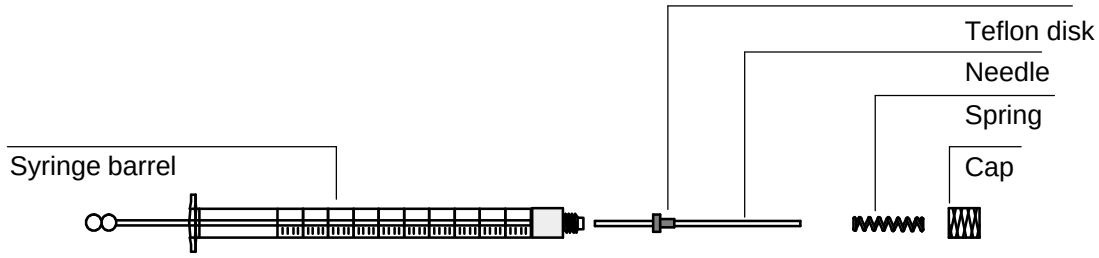
Note: The Teflon ferrule may lose its seal in time. If so, first retighten the retaining nut, and if the seal still leaks, install a new Teflon ferrule and needle.

Replacing the universal syringe needle

The stainless steel needles used for 250 µm and 320 µm injections must be inserted into a glass syringe barrel before they can be used in the HP 7673 Automatic Sampler.

To insert a needle into a syringe barrel, follow the procedure below:

1. Unscrew the syringe barrel cap and remove the spring.
2. Select the correct size needle for the column you plan to use, and make sure the needle has Teflon disk, as shown in the picture below. If the syringe barrel does not have the Teflon disk, use the instructions in the syringe box to wrap the needle yourself.
3. Slide the spring and the cap down over the needle.
4. Insert the needle into the syringe barrel.
5. Screw the cap back on the syringe barrel.



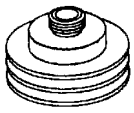
Automatic injection considerations

Syringe needles can be made of stainless steel (size 26 gauge or tapered 26-23 gauge) and made from fused silica (size 530 μm, 320 μm, or 250 μm). To operate using 250 μm and 320 μm needles, proceed to the next page.

When you are performing analyses using an automatic sampler, you must use the disk type septum for 530 μm and larger needles and the through-hole septa for smaller needles. In addition, consider the following:

- ***For HP 18593B injector modules***, remove the needle guide top from the septum nut base assembly. Save the needle guide top to realign the bracket.
- ***For HP 18593A injector modules***, leave the needle guide top on the septum nut base assembly. The cup-shaped top is part of the injector mounting scheme.

Septum nut base assembly



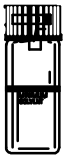
Needle guide top

Automated injection technique

You can automatically inject into 250 μm, 320 μm, 530 μm columns using the on-column inlet However, be sure to align the needle, inlet, and column to ensure the best performance.

Use this checklist to make sure you are ready to inject using the HP 7673 Automatic Sampler. For complete instructions on how to operate and troubleshoot the automatic sampler, see the *HP 7673 Automatic Sampler Manual.*

- ☐ The sample vials are half full.
- ☐ The vial cap is centered, is not wrinkled, and the septum is flat
- ☐ The sample inserts and vials match the run parameters



- ☐ Each solvent bottle contains 4.5 ml of fresh solvent.
- ☐ Waste bottle is clean and empty.
- ☐ You are using 2 waste bottles with the tray.
- ☐ The number of solvent bottles is limited to number of sample vials.

- ☐ The syringe is the correct design and size for your application.
- ☐ The plunger carrier screw is tight.
- ☐ The needle is aligned with the septum retainer nut.

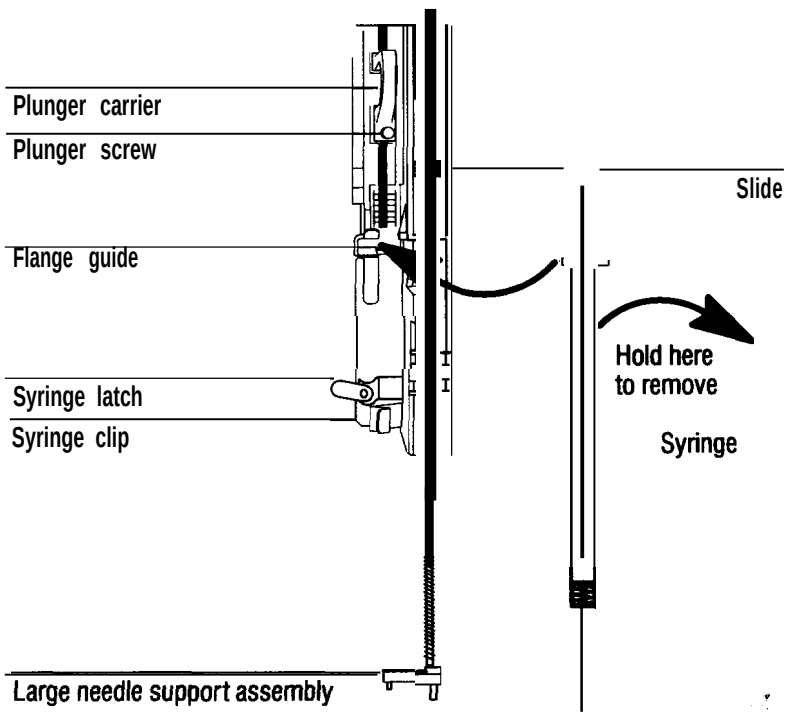
- ☐ You have installed the correct septum type.
- ☐ The septum has been used for fewer than 200 injections.

- ☐ The injector run parameters are set correctly
  - Injection mode matches type of inlet.
  - Number of injections per vial is less than 5.

Performing automated injection onto 250 µm and 320 µm columns

This section lists the proper procedures for changing, aligning, and installing the needle support assembly to adapt from 530 µm injection to 250 µm or 320 µm injections. To inject onto 250 µm or 320 µm columns, you first change the needle support assembly.

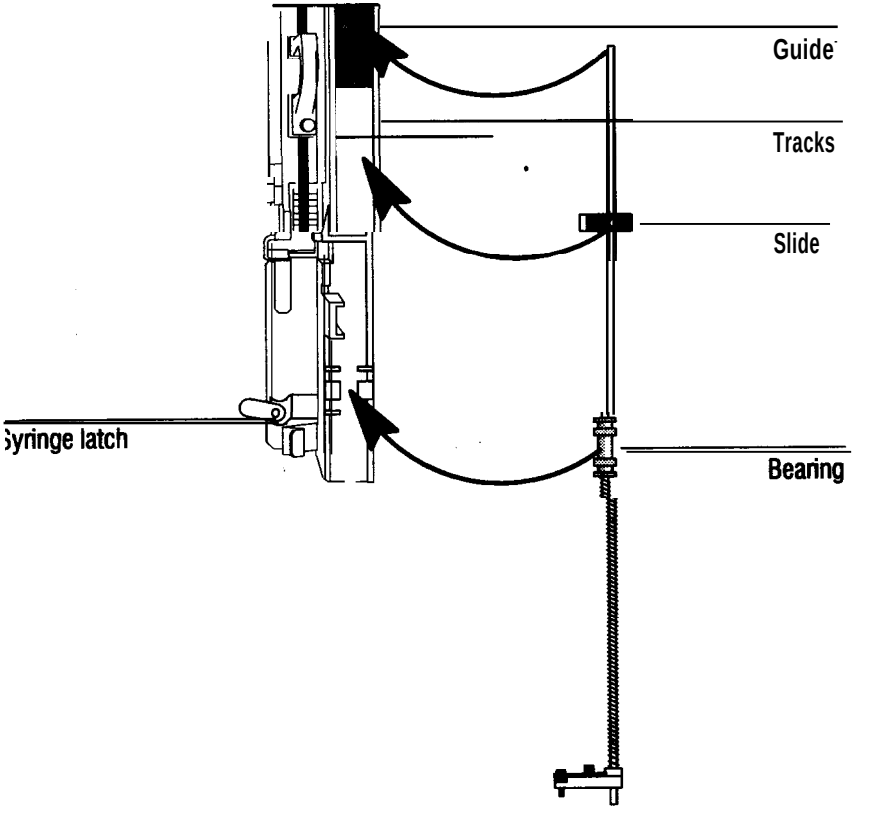
Removing the 530 µm needle support assembly



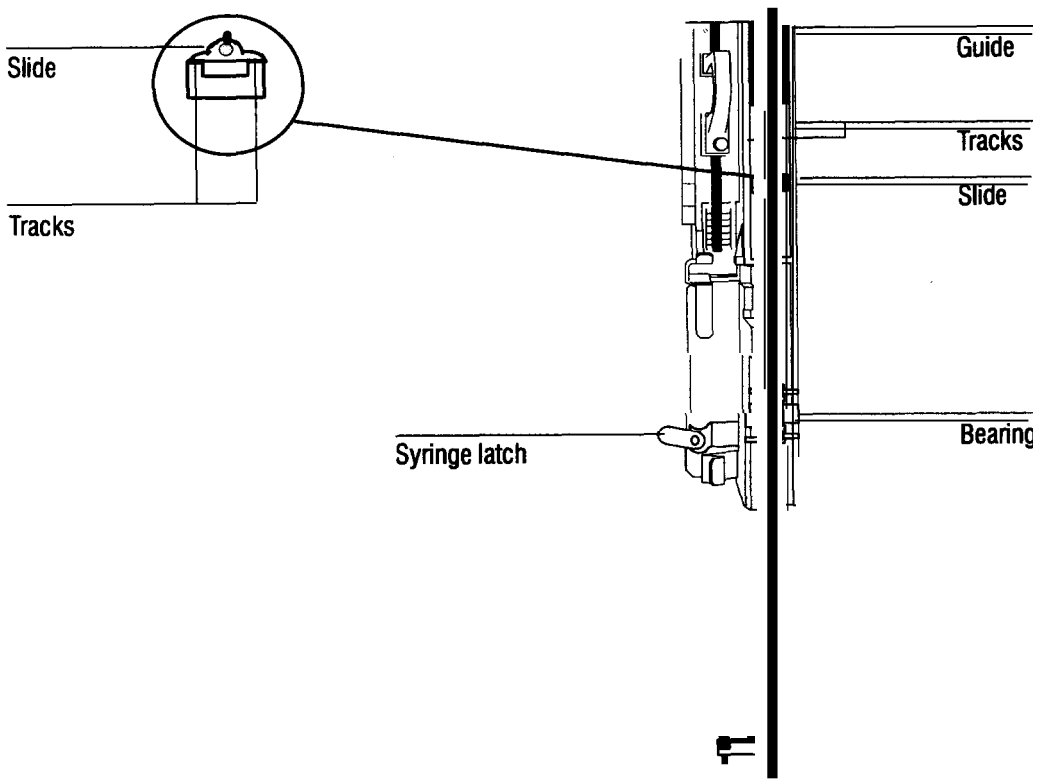
1. Lay the injector module on its back on a flat surface, and open the injector door.
2. Loosen the plunger screw and slide the loop of the plunger carrier up as far as it will go.
3. Swing the syringe latch counterclockwise to unlock the syringe.
4. With your finger under the upper portion of the syringe barrel (just above the syringe latch), pull the syringe up and gently remove it.
5. With your finger under the brass fitting of the 530pm needle support assembly, pull up gently to release and remove the assembly.

Installing the 250 µm and 320µm needle support assembly

1. Holding the new needle support assembly in your right hand, push the bearing down the shaft of the assembly about 3 inches.
2. Insert the upper end of the rod into the black plastic guide to the right of the plunger carrier loop.
3. Align the bearing on the needle support assembly with the plastic bearing clip to the right of the syringe latch.



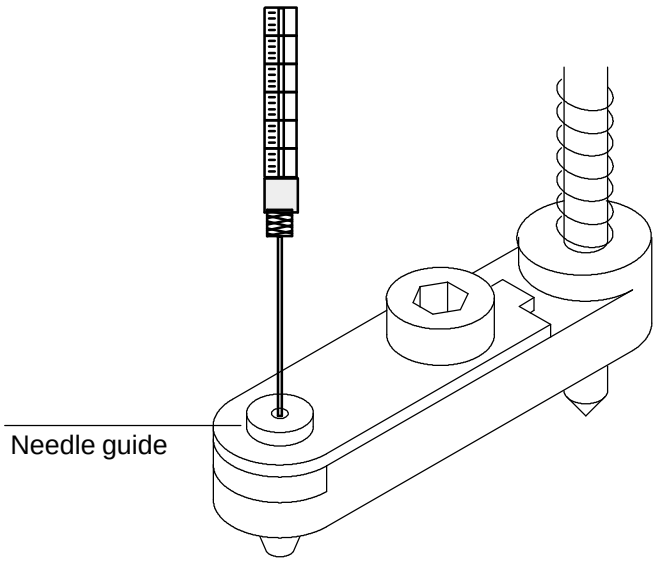
4. Push the assembly down into place. Make sure the slide lies flat on the tracks of the syringe carriage so that it glides up and down as shown in the illustration below.



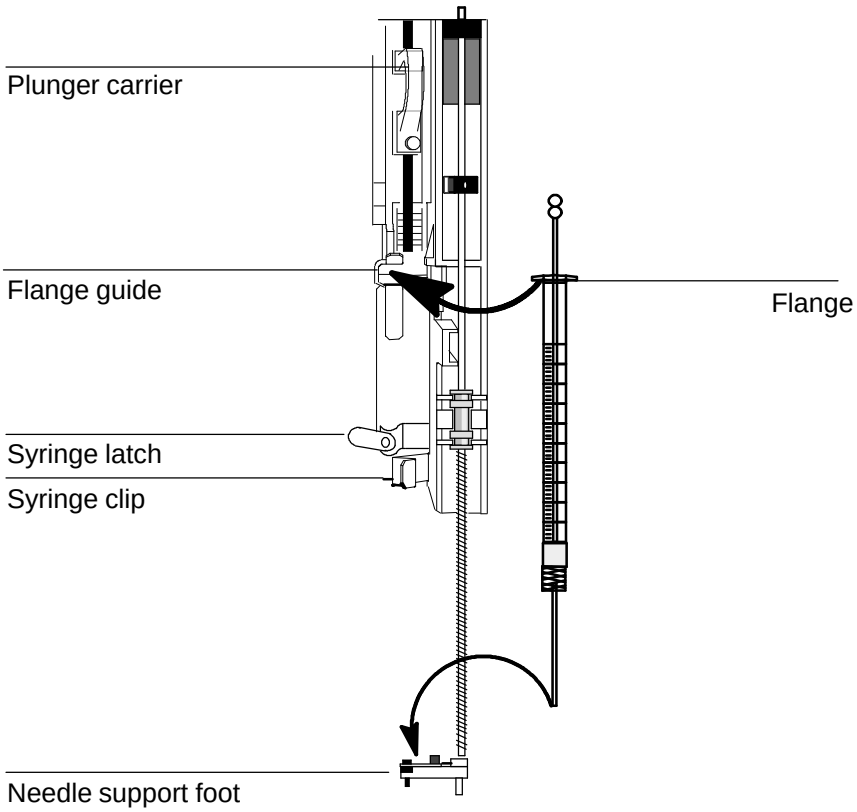
Installing the syringe into the needle support assembly

When a syringe is worn, replace it with a new one using the following steps:

1. If you have removed the injector module to replace the needle support assembly, place the module back on the HP 5890 Series II GC oven or on a parking post if you have one.
2. Open the injector door.
3. Pass the syringe needle through the hole of the small, needle guide in the needle support foot.



4. Align the flange with the flange guide and syringe clip, and gently press the syringe into place, keeping the needle in the hole of the needle guide. Be careful not to bend the needle during this step.
5. Close the syringe latch by swinging it clockwise.



Caution Do not operate the injector without a syringe in place because the syringe latch may interfere with the motor if it is allowed to swing freely.

6. Move the plunger loop down, and tighten the plunger screw.
7. Move the plunger loop up and down to make sure the plunger is moving with the carrier.
8. Make sure the needle is aligned with the needle guide in the foot by moving the slide up and down. The needle should slide smoothly in the needle guide.

Caution Failure to use the correct size on-column syringe when injecting into an on-column inlet could damage the injector, syringe, and column.

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Maintenance and Troubleshooting

Maintenance and Troubleshooting

This section lists procedures to keep your inlet functioning properly, including:

- Cleaning and care
- Pressure and temperature control problems
- Safety shutdown and alarm relay
- Septum problems
- Syringe problems
- Sampler vial cap septum problems
- FID flameout problems
- Proper configuration
- Peak broadening and split peaks
- Useful tools

Cleaning and care

Most laboratories have airborne lint and dust that accumulates on the needle guide and can be carried into 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.

Clean the stainless steel needle guide, spring, and insert by sonication for 1 minute in aqueous detergent, then in distilled water. Rinse with methanol, and air dry. Check the insert for cleanliness with a magnifier.

Clean the fused silica needle guide by forcing methanol through the needle with a squeeze bottle.

Troubleshooting automatic and manual injections

If you have checked these possible causes and still have a problem, call your nearest Hewlett-Packard Service office.

Symptom	Possible Cause	Corrective or Preventive Action
Not enough pressure (safety shutdown activated).	Septum leaks or is missing.	Check system for leaks.
	Column is broken.	
	Column ferrule seal leaks.	
	Gas supply is off.	
	Supply pressure is inadequate.	
	Desired pressure may not be achievable with the column in use.	
Pressure goes to 0 or maximum.	Configuration is wrong.	Check your configuration in “Proper Configuration”.
Not Ready light flickers (oscillating pressure).	Septum or column connection leaks.	
	Pressure set higher than the operating limit.	
Not Ready light flickers (oscillating temperature).	Configuration is wrong.	See “Proper Configuration”.
	Inlet temperature equilibration time is too short.	Increase equilibration time.
Pressure and temperature are not controllable.	Configuration is wrong.	See “Proper Configuration”.
Inlet cools down very slowly.	Fan is either not running or blowing away from inlet.	Check that the fan is running.

Symptom	Possible Cause	Corrective or Preventive Action
Bent needle	Incorrectly installed needle support assembly	Check needle support assembly installation.
	Defective needle	Check each syringe before installation to make sure needle is straight.
	Incorrect insert	Make sure the insert is the correct size for the column and needle you use. Also check that the insert is installed correctly.
	Overcrimped vial caps	See *Capping sample vials* in the *HP 7673 Automatic Sampler Manual* for instructions on crimping vial caps.
	Worn or damaged rubber needle guide	Check the needle guide on the needle support foot every time you change the inlet septum, and replace if necessary.
	Incorrect inlet septum	Use only a 5-mm septum with a through-hole.
	Poor alignment of inlet septum and septum nut	Align the inlet septum and septum nut according to the instructions provided in this manual.
	Incorrect column internal diameter	Check the internal diameter of the column by using the appropriate insert.
	Closed inlet septum hole	Replace the septum.
	Poor alignment of the inlet and the automatic injector	See the *HP 5890 Series II Operating Manual* for alignment instructions.

Symptom	Possible Cause	Corrective and Preventive Action
No peaks or unexpectedly small peaks	Plugged syringe needle	Replace the needle, or clean it with wire.
	Worn syringe barrel	Replace the syringe often, or use a gas-tight syringe. (Under high pressure, the sample is pushed upward through the gap between the plunger and the glass barrel, and a worn plunger can lead to sample loss.)
	Loose removable needle	Make sure syringe barrel caps are screwed on tightly and that the Teflon disk is wrapped tightly.
	Incorrectly placed or missing Teflon disk on the syringe needle	Check every syringe needle to make sure the Teflon disk is present and correctly placed.
Poor precision; poor repeatability; large standard deviation	Worn syringe barrel	Replace the syringe often. (Under high pressure, the sample is pushed upward through the gap between the plunger and the glass barrel, and a worn plunger can lead to sample loss.)
	Loose removable needle	Make sure syringe barrel caps are screwed on tightly and that the Teflon disk is in place.
	Widened holes in vial caps	Replace vial caps when holes widen and leaks develop. (A sample with a low boiling point can escape through a hole that has become too large.)
	Inlet pressure set too low.	Adjust the pressure.
	Incorrectly placed or missing Teflon disk on the syringe needle	Check every syringe needle to make sure the Teflon disk is present and correctly placed.

Safety shutdown

Programmable cool on-column inlets that are equipped with electronic pressure control have a safety shutdown feature to prevent gas leaks from creating a safety hazard. If the system cannot reach a pressure setpoint, the system beeps. After about two minutes, the beeping stops and the following message appears on the display:

Also, a relay signal is set that can trigger an alarm.

A safety shutdown can occur under the following conditions:

1. There is a leak in the system (see *Pressure and temperature control problems*). This includes missing septa or columns!
2. The column is not restrictive enough to reach desired pressure (i.e., 530 µ columns will not go to 100 psi with available flow).

   Note: This may occur during a programmed pressure ramp that is too high a pressure.
3. There is insufficient supply pressure.
4. Configuration is set wrong. Check the mode switch on the inlet controller board (see *Proper configuration*).

To recover from a safety shutdown, turn the GC power off, then on. Then reset temperature and pressure zones to desired values. (After safety shutdown, pressure setpoint is automatically reset to zero.)

Septum problems

The od and thickness of the disk septa are critical for long life and leak-free operation. If the od is too small and the septum is too thick, the septum will fail prematurely. A thick septum can bend needles and make needle insertion difficult.

If the septum drops easily into the retaining well and falls out when the needle guide is turned upright, the od is too small. The od should be 5 mm, and the septum should fit snugly in the retaining well.

When the needle guide is screwed onto the inlet, resistance will be felt when contact is made with the septum. This should occur when the guide is 1/8-or 1/4-turn from bottoming on the inlet body. If contact occurs sooner, the septum is too thick. If no resistance is felt, the septum may be too thin.

Syringe problems

For manual injection with fused silica needles, make sure the needle is clean and free of dust. Wipe with solvent wet tissue, and wipe dry just before injection. Use the same solvent used to dissolve the sample. If the needle should break, replace it. Do not use a needle less than 10 cm long.

For automatic injection, the same cleanliness precautions apply as mentioned earlier. All the areas in the automatic sampler that introduce samples (wash/waste bottles, needle guide) collect dust and should be cleaned periodically.

For longer septum life, you can polish the syringe needle with fine abrasive to remove any concentric ridges left from the needle drawing process. These are visible when magnified, and, if present, can scrape septum particles into the inlet.

Automatic sampler vial cap septum problems

Contamination can occur from vial cap septa, particularly if more than three injections are made from one vial. Repeated punctures may dislodge septum pieces into the sample. To determine if this is a contamination source, cut a 1- x 2-mm segment from a septum and slurry in a vial with 1 ml of the same solvent used for the sample. Analyze this mixture under the same conditions used for sample analysis. If peaks appear that might interfere with your analysis, try other types of septa until you find one compatible with your analysis. A similar test can be run with the injection port septum if you suspect it to be a source of contamination.

Contamination can also occur when small particles are picked up by the sampler syringe and delivered on column. The symptom is sudden onset of a tailing solvent and early eluting component peaks.

The only solution is to remove enough column to eliminate the particles. The easiest way to find them is to backlight the column with a flashlight. Cover the lens with a tissue to diffuse the light. Move the light along the column until the offending particles are found and cut off enough column to eliminate them. Use a magnifier if necessary; a single, very small particle will cause significant tailing.

FID flameout problems

When using pressure programming with large id columns (i.e., 530 µ columns) it is possible to blow out the FID flame if pressure (flow) becomes too high. If this occurs, either lower the pressure ramp or switch to a more restrictive column (longer and/or smaller id).

Peak broadening and split peaks

As a sample is injected on the column at a temperature below the boiling point of the solvent, the carrier gas pushes the liquid farther into the column, creating a flooded zone. The solute components are spread over the length of the flooded zone. This is called *band broadening in space.*

1. Sample on column at moment of injection

2. Carrier gas pushes liquid solvent to create a flooded zone. The solutes distribute themselves evenly across this zone, causing band broadening. The flooded zone is made worse when the solvent is less soluble in the stationary phase.

The length of the flooded zone depends on the solubility of the solvent in the stationary phase. If the solvent is less soluble, the flooded zone will be longer, and peak broadening will be greater.

Further distortion comes from nonuniform distribution of low volatility compounds across the solvent band. This gets worse as sample volume increases, and column length or diameter decreases.

Possible solutions

1. **Check Solubilities.** Make sure the polarity of the solvent is compatible with with polarity of the stationary phase you are using. The length of the flooded zone is affected by the solubility of the solvent in the stationary phase. If the solvent is less soluble, the flooded zone will be longer, and peak broadening will be greater.

2. **Use a Retention Gap (RG).** A retention gap is a deactivated, uncoated (or thinly coated) precolumn in series with the analytical column.

   In general, the length of the retention gap required and type of deactivation depend on injected volume and solvent polarity. A working rule of thumb is to use 0.3-m or 1-m of retention gap per µl injected. For a 3 µl sample, use a 3-m RG. The RG should be wetted by the solvent, which means it should be deactivated with material of like polarity. Fused silica tubing is commercially available in a range of diameters and deactivations.

   Automatic injection requires a 0.53-mm id RG, but the analytical column can be of smaller id.

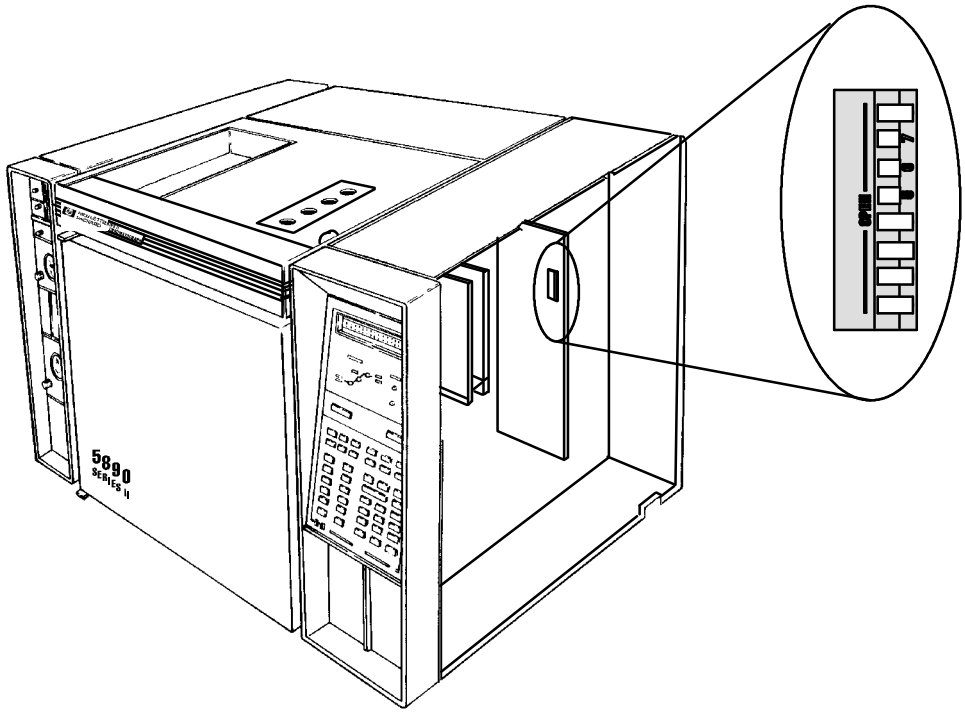
3. **Optimize the Temperature Program and Inlet.** Check the boiling points of the solvent and solutes.

4. **Use the Right Injection Volume.** Know the capacity of the column. Injection volume is important; large volumes may exhibit band broadening, particularly for components whose boiling points are more than 150°C above that of the solvent.

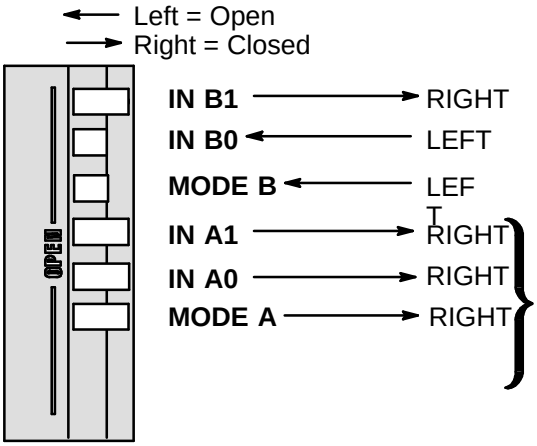
Proper configuration

If the inlet is not working at all, there may be a configuration problem.

1. Turn off the GC power, and remove the side panel of the GC.
2. Check if the switches on the inlet controller board are set for your configuration.
3. Turn on the GC.



Switch setting examples

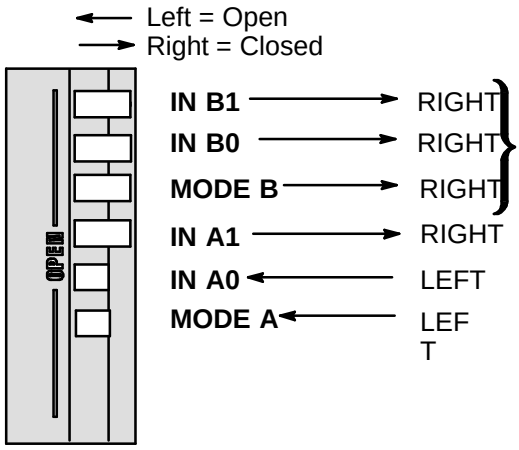


NOTE

This group of switches controls the A position inlet. If another *electronic pressure controlled* inlet is installed in the A position, this group of switches must be set according to the instructions for that particular inlet.

INLET B = Cool On-Column with Electronic Pressure Control

INLET A = Any Non-Electronic Pressure Controlled Inlet

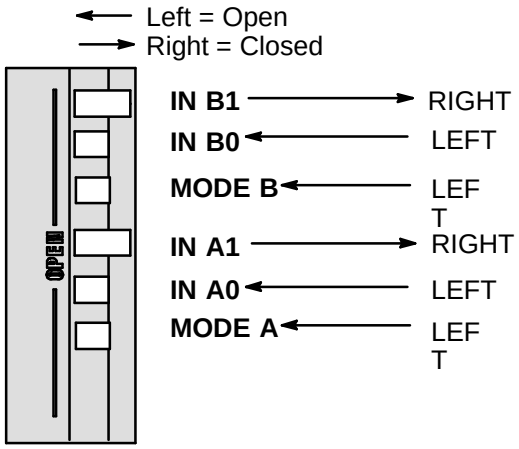


NOTE

This group of switches controls the B position inlet. If another *electronic pressure controlled* inlet is installed in the A position, this group of switches must be set according to the instructions for that particular inlet.

INLET B = Any Non-Electronic Pressure Controlled Inlet

INLET A = Cool On-Column with Electronic Pressure Control



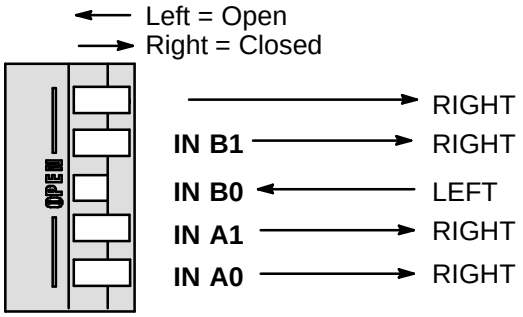
INLET B = Cool On-Column with Electronic Pressure Control

INLET A = Cool On-Column with Electronic Pressure Control

Setting manual pressure control board configuration

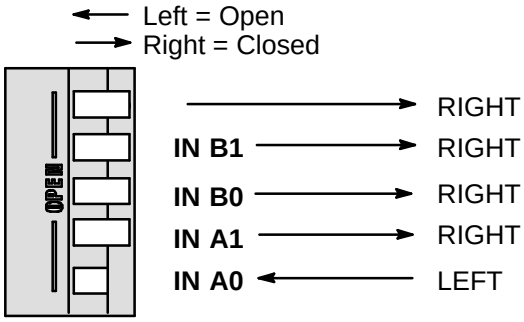
INLET B = Cool On-Column with Manual Pressure Control

INLET A =Any Other Inlet



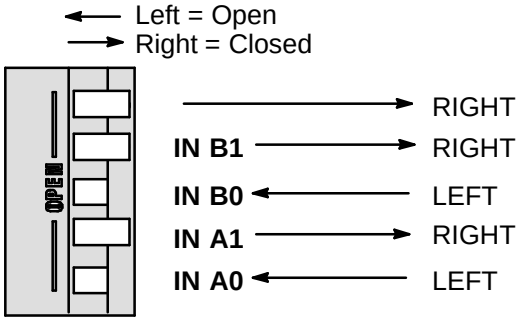
INLET B = Any Other Inlet

INLET A = Cool On-Column with Manual Pressure Control



INLET B = Cool On-Column with Manual Pressure Control

INLET A = Cool On-Column with Manual Pressure Control



Useful tools

- **Ultrasonic cleaning bath•** It is the only effective way to clean small parts.
- **Tungsten carbide knife•** Used for cutting fused silica tubing. The knife edge can be kept sharp with an inexpensive diamond hone, such as those used for touching up carbide-tipped saw blades and router tips.
- **Drill index (#61-80)•** Used for drilling holes in Vespel and Graphite/Vespel ferrules.
- **Reamers•** Used to ream slightly undersized holes drilled in ferrules to match column diameter closely.
- **10x magnifier•** Used for examination of column ends and small hardware.
- **Hemostats** (curved or straight)**•** Very handy for manipulating small parts without losing them or contaminating with fingerprints.
- **Fine abrasive paper** (emery or 600-grit silicon carbide)**•** Used for smoothing rough edges and polishing surfaces.

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