Operating Manual

HP 5890 Series II and HP 5890 Series II Plus

HP part number
05890-90261

First Edition, Jun 1989
Second Edition, Oct 1989
Third Edition, Jan 1990
Fourth Edition, Oct 1990
Fifth Edition, Oct 1991
Sixth Edition, Sep 1992
Seventh Edition, Jun 1993
Eighth Edition, Nov 1993
Ninth Edition, Jul 1994

Printed in USA

Warranty

The information contained in this document is subject to change without notice.

Hewlett-Packard makes no warranty of any kind with regard to this material, including, but not limited to, the implied warranties of merchantability and fitness for a particular purpose. Hewlett-Packard shall not be liable for errors contained herein or for incidental or consequential damages in connection with the furnishing, performance, or use of this material.

Safety Information

The HP 5890 Series II and HP 5890 Series II Plus are IEC (International Electrotechnical Commission) Safety Class 1 instruments. This unit has been designed and tested in accordance with recognized safety standards. Whenever the safety protection of the HP 5890 Series II has been compromised, disconnect the unit from all power sources and secure the unit against unintended operation.

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 horder 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 IS0 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 Maschinenlärminformationsverordnung 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
Hewlett-Packard (Company
2850 Centerville Road
Wilmington, DE 19808-1610

Contents

Chapter 4 — Setting Inlet System Flow Rates 69

Chapter 5 — Operating Detector Systems 95

Chapter 10 — Using Electronic Pressure Control 215

1

Getting Started

Getting Started

Installation checklist

This checklist will help you get your HP 5890 Series II into operation. All references are to the HP 5890 Series II Manual Set.

	Checklist	Location
1.	Select a location for the instrument.	Site Prep and Installation, Chapter 1
2.	Check the new instrument for damage in shipment.	Site Prep and Installation, Chapter 2
3.	Make sure everything is present.	Site Prep and Installation, Chapter 2
4.	Place the instrument in position and make all connections.	Site Prep and Installation, Chapter 2
5.	Turn the instrument on.	Site Prep and Installation, Chapter 2
6.	Install a column.	Operating, Chapter 2
7.	Set the inlet system flow rate.	Operating, Chapter 4
8.	Set appropriate heated zone temperatures.	Operating, Chapter 3
9.	Set the detector system flow rates.	Operating, Chapter 5
10.	Turn the detector on.	Operating, Chapter 5
11.	The instrument is now ready to make a run.	Operating, Chapter 7

Daily startup

1. Check that the operating conditions are correct for your analysis. Make any changes that are needed.
2. Reset the detector sensitivity if you lowered it overnight.
3. If you're using temperature programming, make a blank run (no sample) to clean out any septum bleed or carrier gas impurities that might have accumulated in the column.
4. Start your analysis.

Daily shutdown

1. In most cases, leave the detectors on and at operating temperature. This will avoid a long equilibration time in the morning. You may want to reduce the sensitivity particularly with the TCD and NPD detectors, to prolong their lifetime.
2. Leave the carrier flow on to protect the column(s). For extended shut-down periods, cool the oven to room temperature and then turn the carrier flow off.
3. Now is a good time to change the inlet septum if needed. Volatile material will be baked out overnight. But keep the columns warm so that the baked-out material doesn't accumulate on the column. You can generally expect 1- or 2-days use from a septum, but this is reduced by high temperatures, many injections, dull or hooked needles, etc. It's best to avoid trouble by changing them daily.

Abbreviations

A/D	Analog-to-Digital	LED	Light Emitting Diode
Ar/CH_4	Argon/Methane (5% or 10%)	NPD	Nitrogen-Phosphorus Detector
ECD	Electron Capture Detector	OD	Outside Diameter
EFS	Electronic Flow Sensing	RAM	Random Access Memory
FID	Flame Ionization Detector	ROM	Read-Only Memory
GPV	General Purpose Valve	SCC	Single-Column Compensation
ID	Inside Diameter	S/ECM	Sampler/Event Control Module
INET	Instrument Network	TCD	Thermal Conductivity Detector
LAS	Laboratory Automation System	TTL	Transistor-Transistor Logic

Conversions

1 inch (in.) = 2,54 cm
1 psi (pound per square inch) = 6.89 kPa
1 pound (lb) = 0.454 kg
1 µl = 10^{-6} 1 (Liter)
1 pA = 10^{-12}A (Ampere)
1 mV = 10^{-3}V (Volt)
1 µm = 10^{-6} m (Meter)

HP 5890 signal output (full scale)

(**+1 mV** output, ATTN 2↑() and RANGE 2↑() = 0)

TCD 1 mV, nominal

FID 1 x 1 0^{-12}A

NPD 1 **x 1** 0^{-12}**A**

ECD 10 Hz. Note that under these conditions, 1 mV (1/1000 of full scale) is observed at the + IV output.

General safety considerations

Note: The HP 5890 is a Safety Class I instrument, manufactured and tested according to international safety standards.

There are a number of common sense safety considerations to keep in mind at all times in using the HP 5890:

- H_2 (Hydrogen) is flammable, and explosive when confined in an enclosed volume (for example, the oven). In any application using H_2, turn off the supply at its source when changing columns, performing service procedures, etc.

  When measuring gas flow rates through an FID or NPD, never measure air and H_2 together. They should be measured separately to minimize explosion hazard.

- The HP 5890 is supplied with a three-conductor power cord providing a protective earth ground connection when plugged into a properly wired receptacle. Proper receptacle grounding must be verified.

- The oven, inlet, and detector zones may be hot enough to cause burns. Connected hardware also may be sufficiently hot enough to cause burns. If necessary, heated areas maybe turned off and allowed to cool.

- To avoid shock hazard, the power line cord must be disconnected at its receptacle anytime the rear cover panel must be removed. Also, connected devices should also be disconnected at their respective line power receptacles.

- An ECD, if installed, must have its exhaust vent connected via external tubing to a proper fume hood.

- Likewise, for a split/splitless capillary inlet system operated in split mode, or for a split-only capillary inlet system, the split vent should be connected via external tubing to a proper fume hood if toxic materials are analyzed, or if H_2 is used as carrier gas.

2

Installing Columns

Installing Columns

The HP 5890 Series II and Series II Plus (hereafter referred to as HP 5890) provide flexibility in choices among inlets, columns, and detectors through use of liners and adapters, allowing any standard column to be used without sacrificing performance. Additional flexibility is gained through positions of inlets and detectors relative to each other and through the large internal volume of the oven.

Note: The Series 530 µ columns supplied with the HP 5890 must be conditioned before use. This is done by establishing a flow of carrier gas at 30 to 60 ml/min through the column while the column is heated at 250° C for at least 4 hours. See *Preventive Maintenance* in the *HP 5890 Series II Reference Manual* for more information about conditioning columns.

New columns should be conditioned because they often contain volatile contaminants absorbed from the air. It may also be necessary to condition a used column that has been stored for some time without end caps or plugs to exclude air.

This section provides information required for proper column installation:

- Liners and inserts
- Preparing packed columns
- Installing packed columns
- Preparing capillary columns
- Installing capillary columns

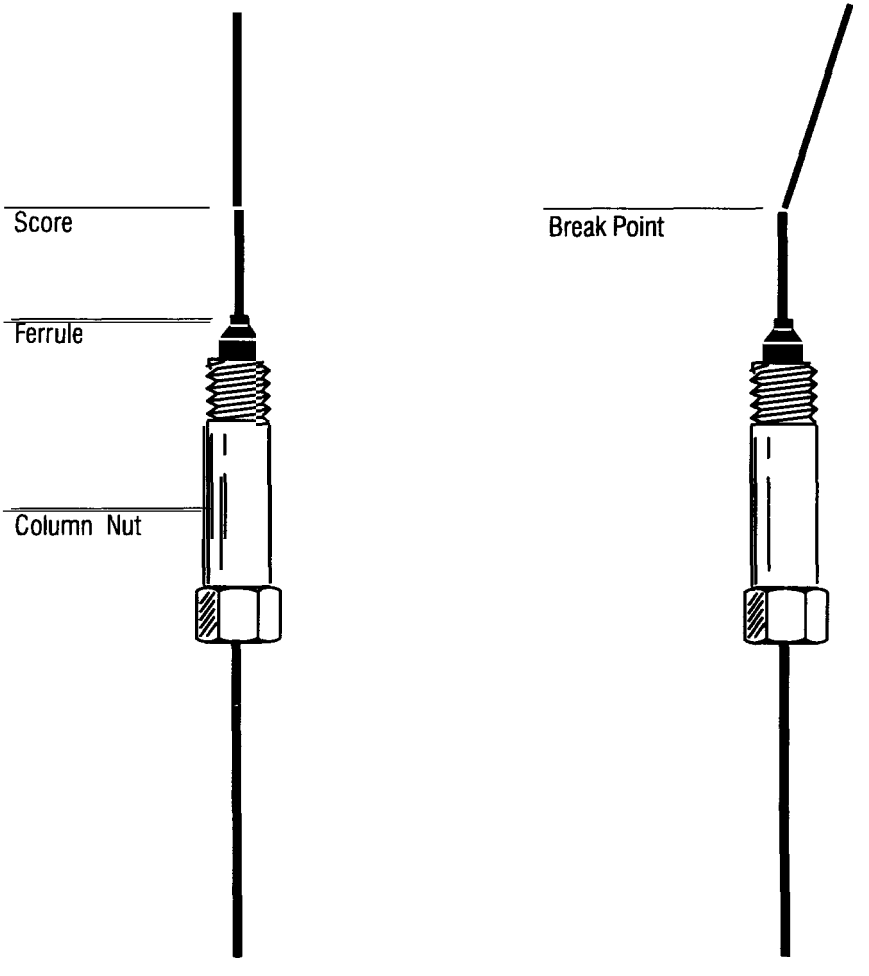
Preparing fused silica capillary columns

Fused silica columns are inherently straight, so no straightening procedures are necessary. It **is** important, however, to have fresh ends of the column, free of burrs, jagged edges, and/or loose particles of column, stationary phase, and/or material from a sealing ferrule or O-ring.

Therefore, whenever the column must be cut to provide fresh ends, use a suitable glass scribing tool (HP ceramic column cutter, part number 5181-8836) to first score the column at the point at which it is to be broken. This is done normally **after** installing on the column, the column nuts and ferrule (or O-ring) required for installation.

WARNING Wear safety glasses to prevent possible eye injury from flying particles while handling, cutting, or installing glass or fused silica capillary columns. Also observe caution in handling capillary columns to prevent skin puncture wounds.

Because of their greater relative rigidity these precautions are especially important in handling Hewlett-Packard series 530 µ capillary columns.



Preparing Fused Silica Capillary Columns

Installing split/splitless capillary inserts

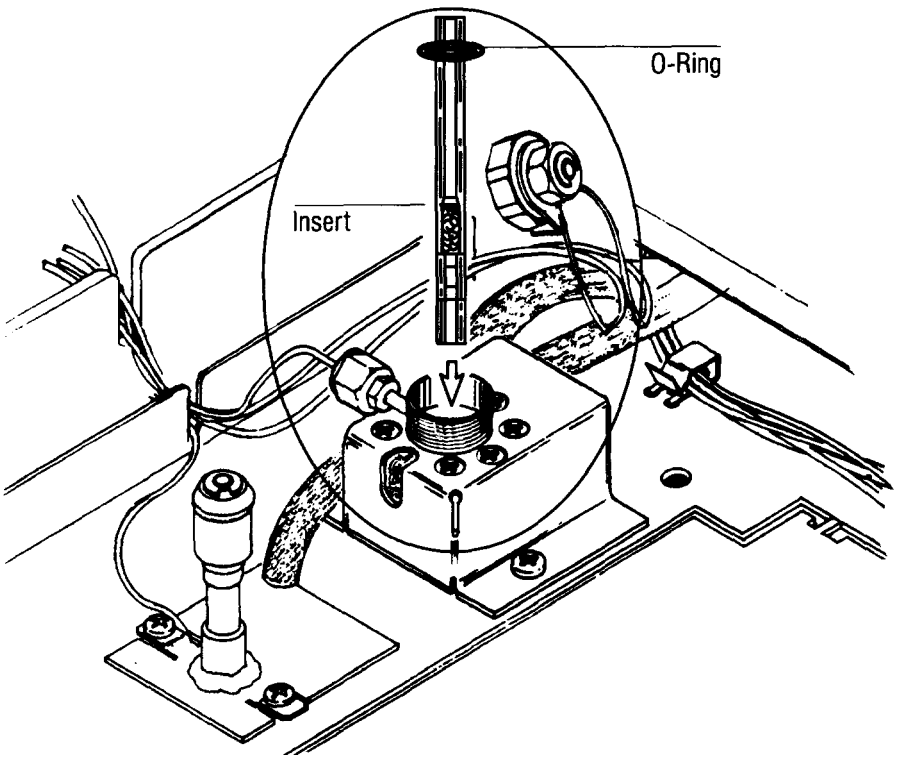
A specific inlet insert is required, depending upon the particular sampling mode. Specific sampling modes include:

- Split, for major-component analyses
- Purged splitless, for trace-component analyses

WARNING Exercise care! The oven, and/or inlet, or detector fittings maybe hot enough to cause burns.

Caution If operating in split mode, carrier gas pressure must be reduced before opening the inlet. If not done, pressure may blow insert packing out of the inlet, altering its characteristics. Pressure is reduced at the column head pressure regulator for the inlet.

1. In handling the insert, avoid contaminating its surface (particularly its interior).
2. Remove the insert retainer nut. The septum retainer nut need not be removed from the insert retainer nut assembly.
3. Using tweezers, forceps, or similar tool, remove any insert already in place.
4. Inspect the new insert to be installed: For a split mode insert, the end with the mixing chamber and packing is inserted first into the inlet.
5. Place a graphite or silicone O-ring on the insert, about 2 to 3 mm from its top end.
6. Replace the insert retainer nut, tightening it to **firm** finger-tightness to form a leak-free seal. Do not overtighten.



Installing Split/Splitless Capillary Inserts

Preparing packed metal columns

Packed metal columns are installed similarly at both the inlet and detector. To minimize unswept (dead) volume column inside the inlet or detector fitting, it is recommended that ferrule(s) be preset and locked onto the column such that the end of the column is approximately flush with the end of the front ferrule (see the top of the figure on page 22).

If not already installed, follow the procedure below to install **new** swage-type nut and ferrules. For metal columns with ferrules already installed and set, proceed to instructions for installing metal columns in this section.

To insure the correct column position, a spacer maybe made from a piece of Teflon tubing:

1. According to the column to be installed (1/8 or 1/4 inch), secure an appropriate **new** male swage-type fitting in a bench vise.

2. Slide a **new,** brass, swage-type nut, rear ferrule, and front ferrule onto a piece of Teflon tubing (1/8 or 1/4 inch). If necessary, use a razor or sharp knife to cut the end of the tubing to present a flat, smooth end.

3. Fully insert the Teflon tubing, ferrules, and nut into the vise-held swage-type fitting. Tighten the nut 3/4-turn past finger-tight to set the ferrules on the tubing. Then remove the assembly from the male fitting.

4. Using a razor knife, cut off the end of the tubing extending beyond the front-most ferrule. Insert the piece into the vise-held swage-type fitting.

   This piece of tubing is now a spacer, insuring that when new ferrules are set onto a column, the column end will be correctly positioned with respect to the end of the front-most ferrule. The male fitting and spacer should be kept on hand to be used whenever new ferrules are being installed on a column.

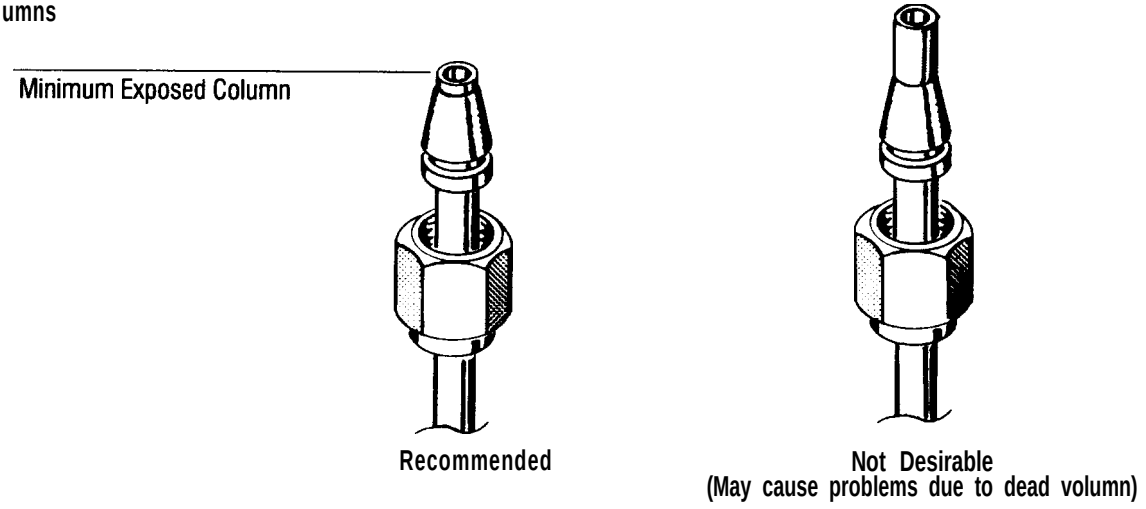
5. Install a **new** swage-type nut and ferrule(s) onto the column.

6. Fully insert the column with its nut and ferrules into the vise-held fitting.

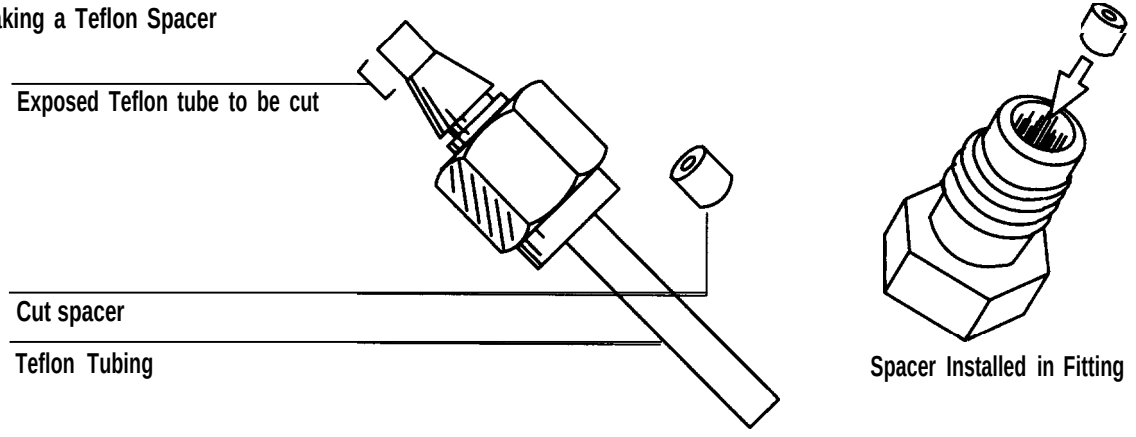
7. First tighten the nut finger-tight. Use a wrench to tighten the column nut an additional 1- and -1/4 turns for l/4-inch columns, or 3/4-turn for l/8-inch columns.

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

Recommended Location for Ferrules on Packed Columns



Making a Teflon Spacer



Preparing Packed Metal Columns

Installing 1/4- and l/8-inch metal columns in packed inlets

Using the figures on the next page as a guide:

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

2. Insert the adapter straight into the inlet base as far as possible.

3. Holding the adapter in this position, tighten the nut finger-tight.

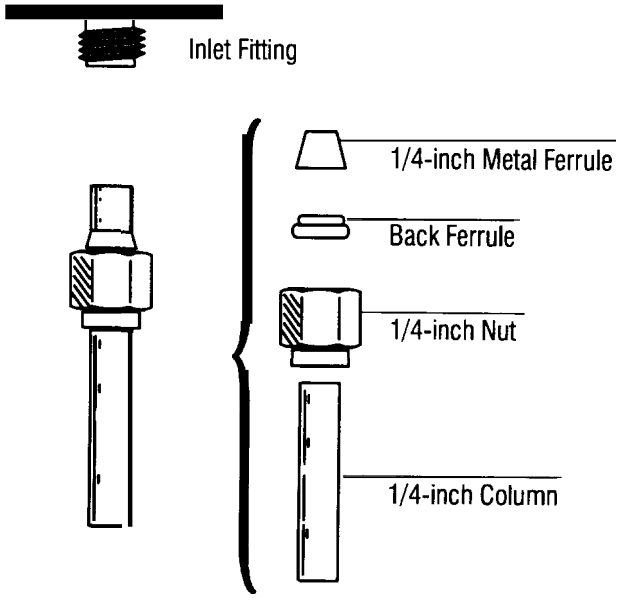
4. Use a wrench to tighten the nut an additional l/4-turn.

To install new swage-type nut and ferrules on the column, follow the procedure *Preparing packed metal columns,* earlier in this section. For metal columns with ferrules already installed, continue with step 5.

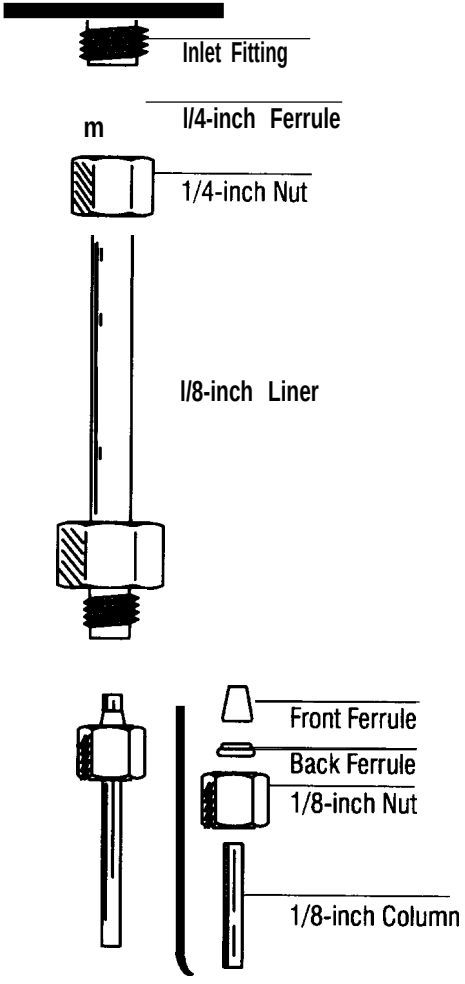
5. Install the column into the inlet by tightening the column nut, assuming ferrule(s) are already set (locked) onto the column (see *Preparing packed metal columns* in this section). Generally an additional l/4-turn past finger-tight is sufficient for l/8-inch columns. For l/4-inch columns, an additional 3/4-turn is usually suffcient.

Use two wrenches in opposition, one on the column nut and the other on the liner body, to prevent rotation of the liner while tightening the column nut.

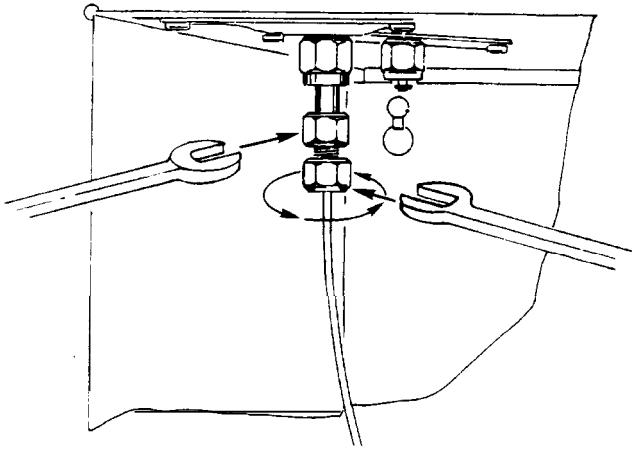
l/4-inch Metal Column, Packed Inlet



l/8-inch Metal Column, Packed Inlet



Using Two Wrenches in Opposition to Tighten Column Fittings



Installing 1/4 and l/8-inch Metal Columns in Packed Inlets

Installing l/4-inch glass columns in packed inlets

At the inlet end, there must be enough column left empty to prevent an inserted syringe needle from contacting either the glass wool plug or column packing (at least 50 mm).

At the detector end, there must be at least a 40-mm empty section to prevent the bottom end of the jet from touching either column packing or glass wool plug.

Because they are rigid, l/4-inch packed glass columns must be installed simultaneously at both the inlet and the detector. The procedure is identical at either end. For information on detector column installation, refer to the appropriate section depending on the detector being used.

Glass columns can be installed with either O-rings or nonmetallic ferrules. For O-ring installation, we recommend using one O-ring with a front metal ferrule, reversed to provide a flat surface for it to seal against.

Using the figures on the next page as a guide:

1. Assemble a brass nut, reversed metal ferrule, and O-ring onto both ends of the column.

   If desired, an extra O-ring maybe placed on the column before the nut. This protects the column by preventing the nuts from dropping into the coiled portion of the column.

2. Insert the column into both the inlet and detector as far as possible. To clear the floor of the oven, it maybe necessary to start the longer end of the column into the inlet at a slight angle.

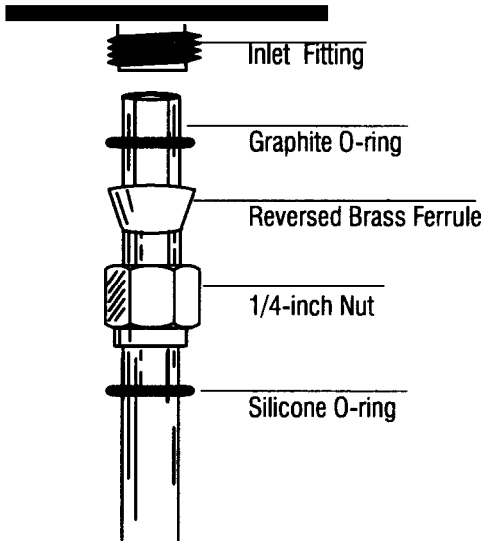
3. Withdraw the column about 1 to 2 mm and tighten both column nuts finger-tight; the degree to which the nut is tightened further depends upon the type of ferrule used:

   - For O-rings, finger-tight is usually sufficient.
   - For Vespel or graphite ferrules, raise the inlet, detector, and oven to operating temperature, then use a wrench to tighten an additional l/2-turn. Tighten further as necessary to prevent leakage.

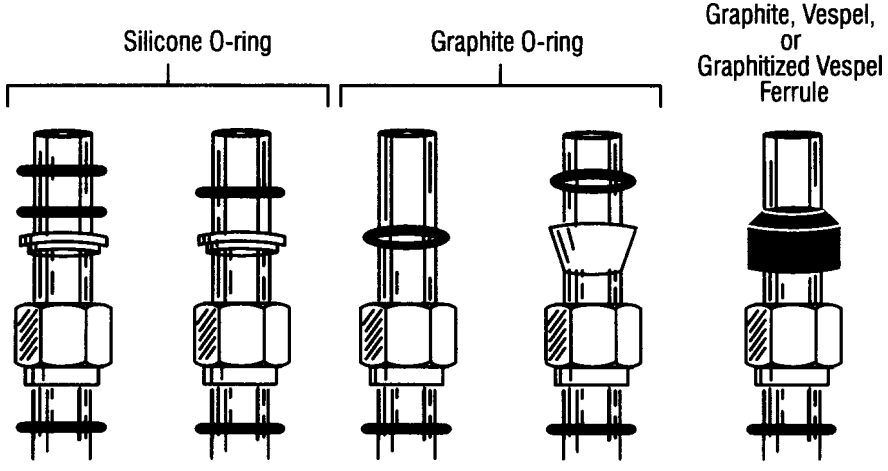
Caution Overtightening the column nut may shatter the column.

l/4-inch Column, Packed Inlet

Recommended



Alternative Installation Methods



Installing l/4-inch Glass Columns in Packed Inlets

Installing capillary columns in packed inlets

Using the figures on page 29 as a guide:

1. Assemble a brass nut and graphite ferrule onto the liner/adapter.
2. Install a glass insert into the liner/adapter.
3. Insert the liner/adapter straight into the inlet as far as possible.
4. Holding the liner/adapter in this position, tighten the nut finger-tight.
5. Use a wrench to tighten the nut an additional l/4-turn.

   Hewlett-Packard capillary columns are wound on wire frames and mount on a pair of brackets that slip into slots at the top of the oven interior.

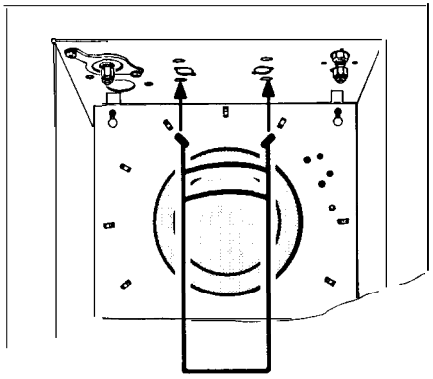
   The bracket has two positions from which to hang the column wire frame. Depending upon frame diameter, use the position that best centers the column in the oven. Column ends should come off the bottom of the frame, making smooth curves to inlet and detector fittings. Avoid allowing any section of the column itself to come in contact with oven interior surfaces.

6. Install on the column, a column nut and ferrule. Note that either a 1. O- or 0.5-mm id graphite ferrule maybe used depending upon column outer diameter.

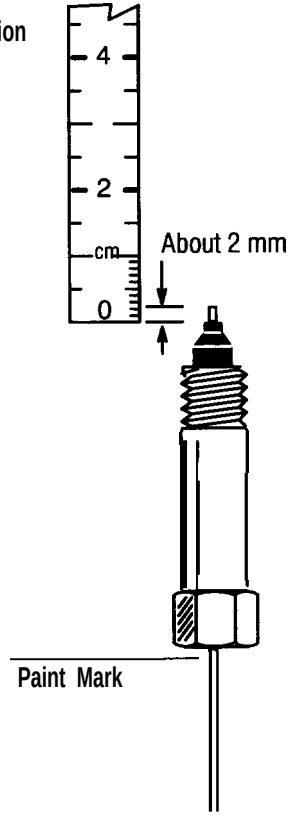
   Inserting the column through the nut and ferrule may contaminate the end of the column. Prepare a fresh column end by following the instructions given in *Preparing fused silica capillary columns* in this section.

7. Position the column so it extends less than 2.0 mm from the end of the ferrule and column nut (threaded end). Mark the column at a point even with the bottom of the nut (hexagonal end). Typewriter correction fluid is a good marking material.
8. Insert column, ferrule, and nut straight into the inlet base. While maintaining the mark on the column so as to be even with the bottom of the column nut, tighten the nut to finger-tightness, then l/4-turn more using a wrench.
9. While holding the spring to the right, slide the capillary liner insulation cup up over the capillary nut. The insulation at the top of the cup should fit flush against the roof of the oven.
10. Release the spring into the groove in the inlet liner.

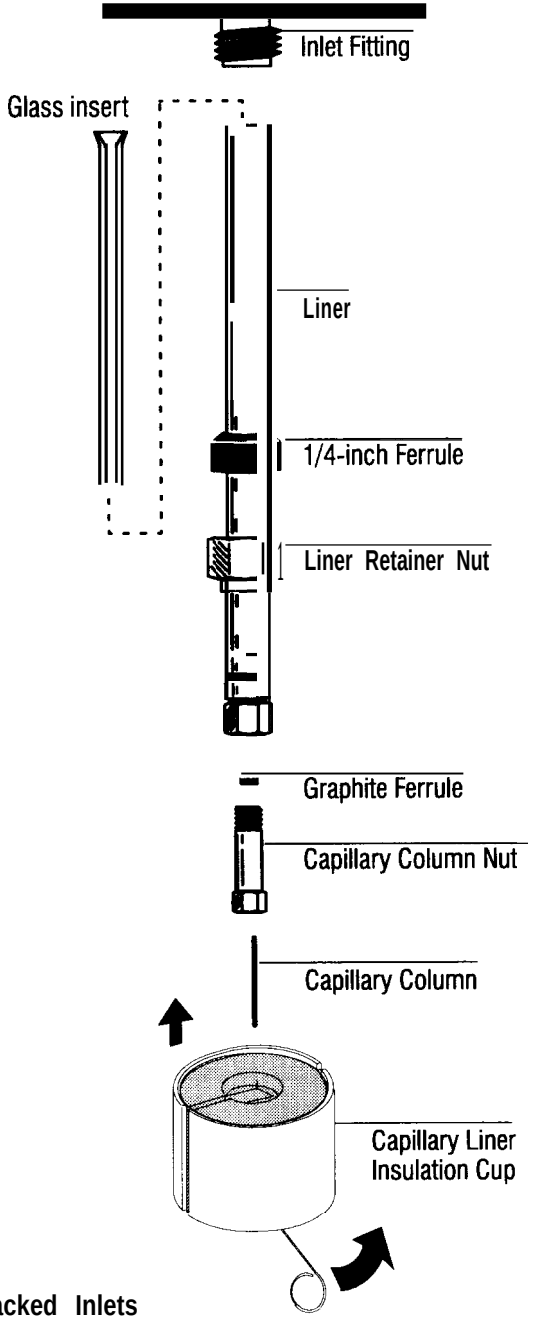
Column Hanger Position



Marking the Column Position



Capillary Column, Packed Inlet



Installing Capillary Columns in Packed Inlets

Installing capillary columns in split/splitless capillary inlets

A specific inlet insert is required depending upon the particular sampling mode, split or splitless. See *Installing split/splitless capillary inlet inserts* in the this chapter, if not already installed.

The following installation procedure assumes that the inlet is prepared properly to receive the capillary column (e.g., that the correct insert is already installed).

Hewlett-Packard capillary columns are wound on wire frames and mount on a pair of brackets that slip into slots at the top of the oven interior.

The bracket has two positions from which to hang the column wire frame. Depending upon frame diameter, use the position that best centers the column in the oven. Column ends should come off the bottom of the frame, making smooth curves to inlet and detector fittings. Avoid allowing any section of the column itself to come in contact with oven interior surfaces.

Using the figures on the next page as a guide:

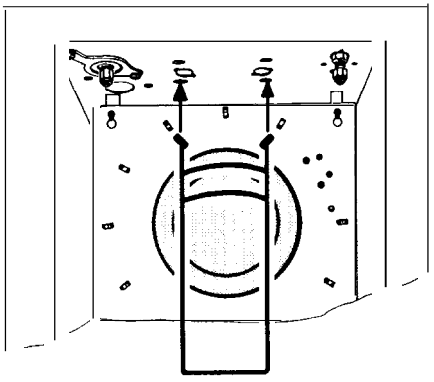
1. Install on the column a column nut and ferrule. Note that either a 1. O- or 0.5-mm id graphite ferrule may be used depending upon column outer diameter.

   Inserting the column through the nut and ferrule may contaminate the end of the column. Prepare a fresh column end following instructions given in *Preparing fused silica capillary columns* in this section.

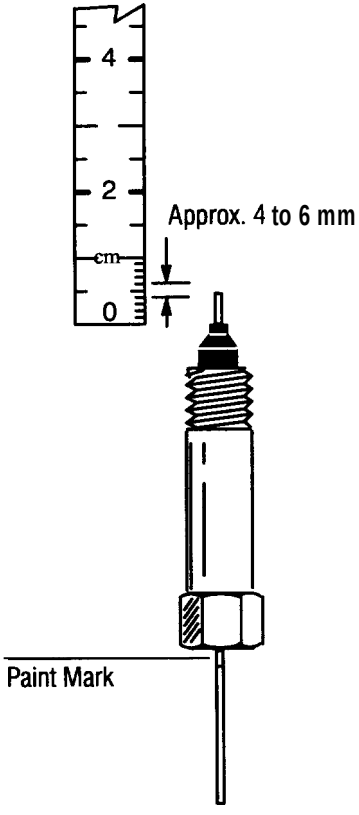
2. Position the column so it extends approximately 4 to 6 mm from the end of the ferrule and column nut (threaded end). Mark the column at a point even with the bottom of the nut (hexagonal end). Typewriter correction fluid is a good marking material.

3. Insert column, ferrule, and nut straight into the inlet base. While maintaining the mark on the column so as to be even with the bottom of the column nut, tighten the nut to finger-tightness, then l/4-turn more using a wrench.

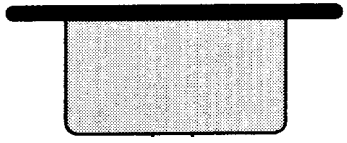
Column Hanger Position



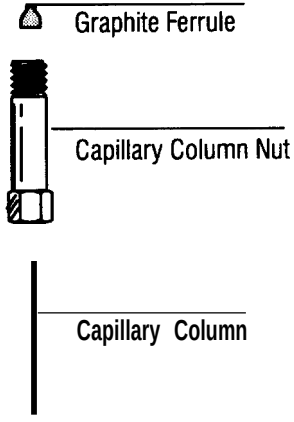
Marking the Column Position



Capillary Column Split/Splitless Capillary Inlet



Inlet Fitting



Installing Capillary Columns in Split/Splitless Capillary Inlets

Installing l/4-inch metal columns in FID's and NPD's

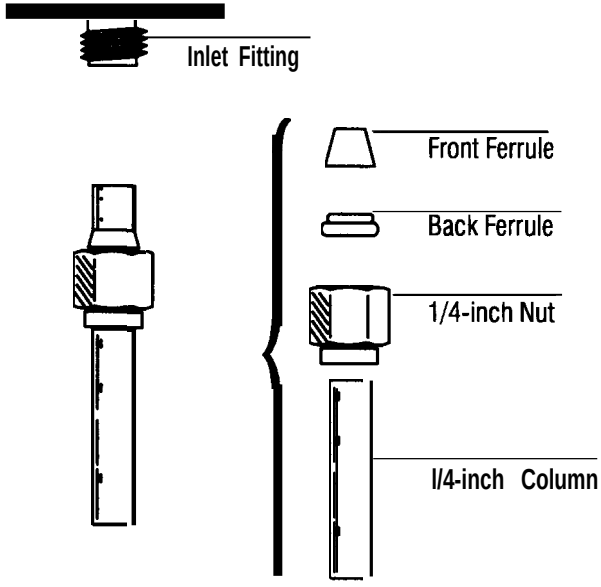
Nitrogen-phosphorus detectors: To avoid contamination of the active element upon receipt, do not remove the seals until ready to connect the column and operate the detector. Failure to observe this simple procedure may reduce the collector's effectiveness or possibly ruin the active element.

To install new swage-type nut and ferrules on the column, follow the procedure *Preparing packed metal columns,* earlier in this section. For metal columns with ferrules already installed, continue.

Using the figures below as a guide:

Install the column into the inlet by tightening the column nut, assuming ferrule(s) are already set (locked) onto the column (see *Preparing packed metal columns* in this chapter). Generally an additional 3/4-turn is usually sufficient.

l/4-inch Packed Metal Column, FID/NPD



Installing l/4-inch Metal Columns in an FID and NPD

Installing l/8-inch metal columns in FID's and NPD's

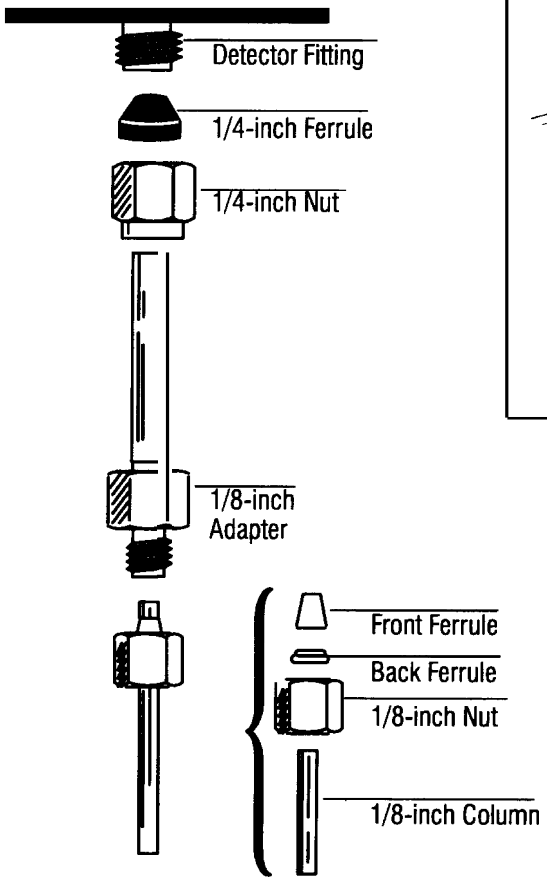
Nitrogen-phosphorus detectors: To avoid contamination of the active element upon receipt, do not remove the seals until ready to connect the column and operate the detector. Failure to observe this simple procedure may reduce the collector's effectiveness or possibly ruin the active element.

Using the figures on the next page as a guide:

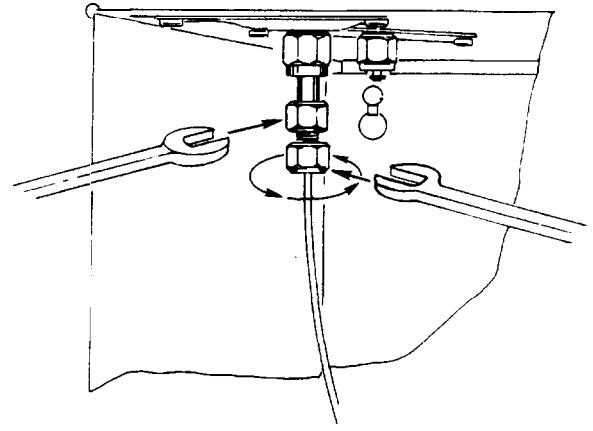
1. Assemble a brass nut and graphite ferrule onto the liner/adapter.
2. Insert the adapter straight into the detector base as far as possible.
3. Holding the adapter in this position, tighten the nut finger-tight.
4. Use a wrench to tighten the nut an additional l/4-turn.
5. Install the column into the inlet by tightening the column nut, assuming ferrule(s) are already set (locked) onto the column (see *Preparing packed metal columns* in this section). Generally an additional l/4-turn is usually sufficient.

   Use two wrenches in opposition, one on the column nut and the other on the liner body, to prevent rotation of the liner while tightening the column nut.

l/8-inch Packed
Metal Column, FID/NPD



Using Two Wrenches
in Opposition to Tighten
Column Fittings



Installing l/8-inch Metal Columns in an FID and NPD

Installing capillary columns in FID's and NPD's

Nitrogen-phosphorus detectors: To avoid contamination of the active element upon receipt, do not remove the seals until ready to connect the column and operate the detector. Failure to observe this simple procedure may reduce the collector's effectiveness or possibly ruin the active element.

Assuming that the 0.011-inch capillary jet (HP part no. 19244-80560) is installed (if not, see Chapter 9, *Preventive Maintenance* in the *HP 5890 Reference Manual),* proceed as follows:

Using the figures on the next page as a guide:

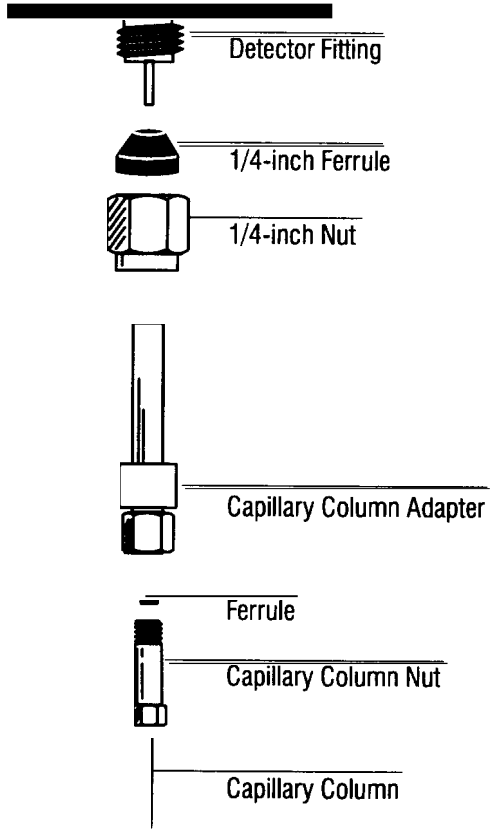
1. Assemble a brass nut and graphite ferrule onto the liner/adapter.
2. Insert the adapter straight into the detector base as far as possible.
3. Holding the adapter in this position, tighten the nut finger-tight.
4. Use a wrench to tighten the nut an additional l/4-turn.
5. Install on the column, a column nut and ferrule. Note that either a 1. O- or 0.5-mm id graphite ferrule may be used depending upon column outer diameter.

   Inserting the column through the nut and ferrule may contaminate the end of the column. Prepare a fresh column end following instructions given in *Preparing fused silica capillary columns* in this chapter.
6. **Gently** insert the column as far as possible into the detector (about 40 mm) until it **bottoms;** do not attempt to force it further. Follow it with the ferrule and column nut.
7. Tighten the nut finger-tight, withdraw the column approximately 1 mm, and then tighten the nut an additional l/4-turn with a wrench.
8. Leak-test the installation at the column nut, both at ambient temperature and with the oven, inlet(s), and detector(s) at operating temperatures. If necessary, tighten fitting(s) further only enough to stop leakage.

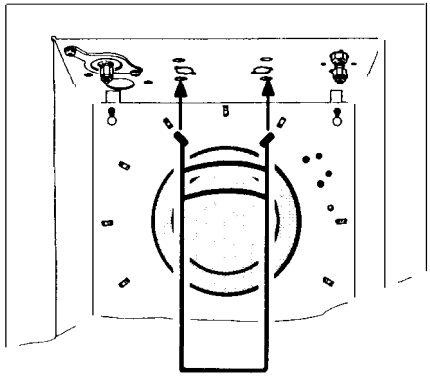
Caution

Leak-detection fluids often leave contaminating residues. After each application, the area checked should be rinsed with CH_3OH (methanol) and allowed to dry.

Capillary Column, FID/NPD



Column Hanger Position



Installing Capillary Columns in FID and NPD

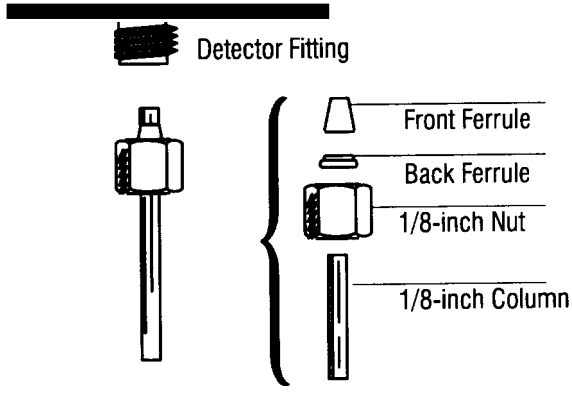
Installing an l/8-inch metal column in a thermal conductivity detector

To install new swage-type nut and ferrules on the column, follow the procedure *Preparing packed metal columns,* earlier in this chapter. For columns with ferrules already installed, continue.

Using the figure below as a guide:

Install the column into the inlet by tightening the column nut, assuming the ferrule(s) are already set onto the column. An additional l/4-turn is usually sufficient.

l/8-inch Packed
Metal Column, TCD



Installing a l/8-inch Metal Column in a Thermal Conductivity Detector

Installing a capillary column in a thermal conductivity detector

Using the figures on the next page as a guide:

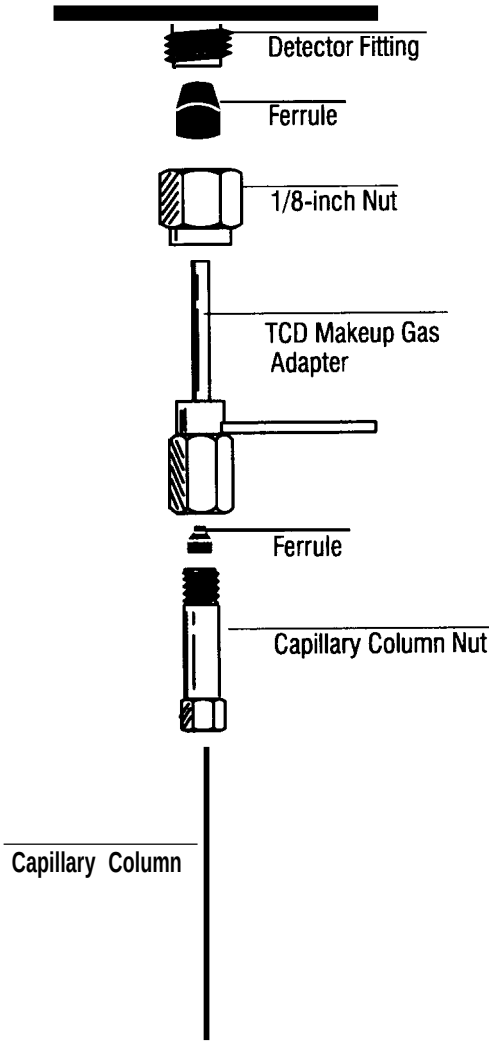
1. Assemble a brass nut and graphite ferrule onto the liner/adapter.
2. Insert the adapter straight into the detector base as far as possible.
3. Holding the adapter in this position, tighten the nut finger-tight.
4. Use a wrench to tighten the nut an additional l/4-turn.
5. Install on the column, a column nut and ferrule. Note that either a 1. O- or 0.5-mm id graphite ferrule maybe used depending upon column outer diameter.

   Inserting the column through the nut and ferrule may contaminate the end of the column. Prepare a fresh column end following instructions given in *Preparing fused silica capillary columns* in this chapter.
6. **Gently** insert the column as far as possible into the detector until it **bottoms;** do not attempt to force it further. Follow it with the ferrule and column nut.
7. Tighten the nut finger-tight, withdraw the column approximately 1 mm, and then tighten the nut an additional l/4-turn with a wrench.
8. Leak-test the installation at the column nut, both at ambient temperature and with the oven, inlet(s), and detector(s) at operating temperatures. If necessary, tighten fitting(s) further only enough to stop leakage.

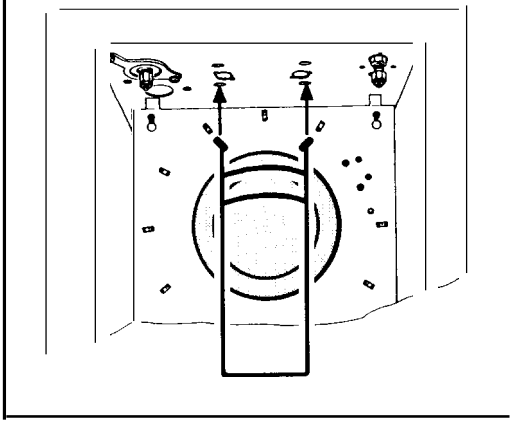
Caution

Leak-detection fluids often leave contaminating residues. After each application, the area checked should be rinsed with CH_3OH (methanol) and allowed to dry.

Capillary Column
With Makeup Gas, TCD



Column Hanger
Position



Installing a Capillary Column in a Thermal Conductivity Detector

Installing a l/4-inch glass column in an electron capture detector

Because they are rigid, l/4-inch packed glass columns must be installed simultaneously at both the inlet and the detector. The procedure is identical at either end. For information on inlet column installation, refer to the appropriate chapter depending on the inlet being used.

Glass columns can be installed with either O-rings or nonmetallic ferrules. For O-ring installation, we recommend using one O-ring with a front metal ferrule, reversed to provide a flat surface for it to seal against.

Using the figures on the next page as a guide:

1. Assemble a brass nut, reversed metal ferrule, and O-ring onto both ends of the column.

   If desired, an extra O-ring maybe placed on the column before the nut. This protects the column by preventing, the nuts from dropping into the coiled portion of the column.

2. Insert the column into both the inlet and detector as far as possible. To clear the floor of the oven, it may be necessary to start the longer end of the column into the inlet at a slight angle.

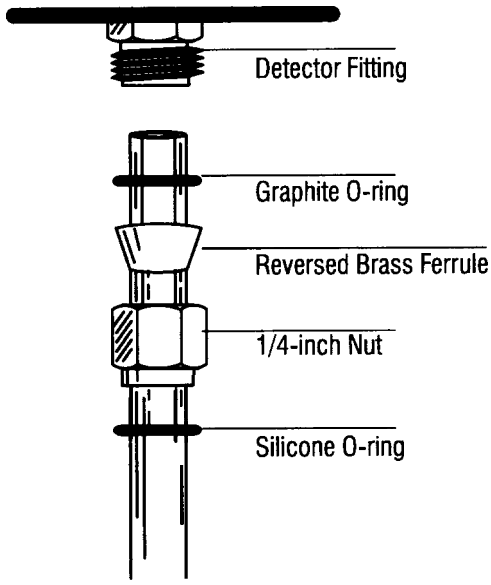
3. Withdraw the column about 1 to 2 mm and tighten both column nuts finger-tight; the degree to which the nut is tightened further depends upon the type of ferrule used:

   - For O-rings, finger-tight is usually sufficient.
   - For Vespel or graphite ferrules, raise the inlet, detector, and oven to operating temperature, then use a wrench to tighten an additional l/2-turn. Tighten further as necessary to prevent leakage.

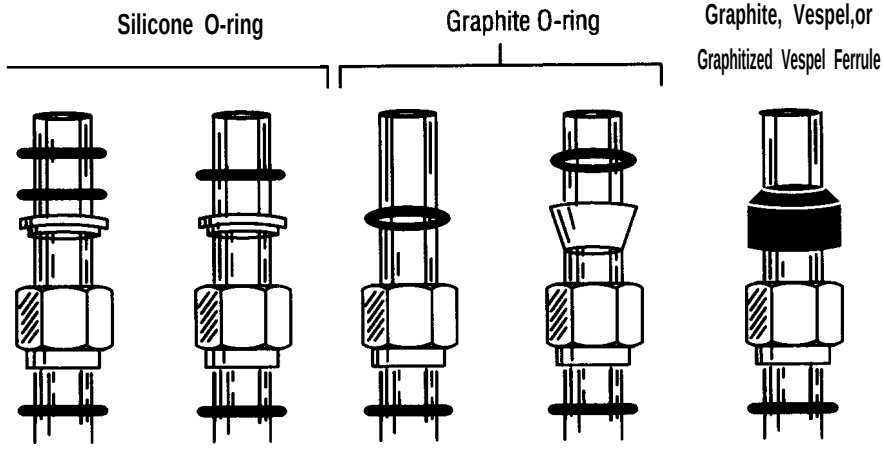
Caution Overtightening the column nut may shatter the column.

l/4-inch Packed Glass Column, ECD

Recommended



l/4-inch Packed Glass Columns, Alternative Installation Methods



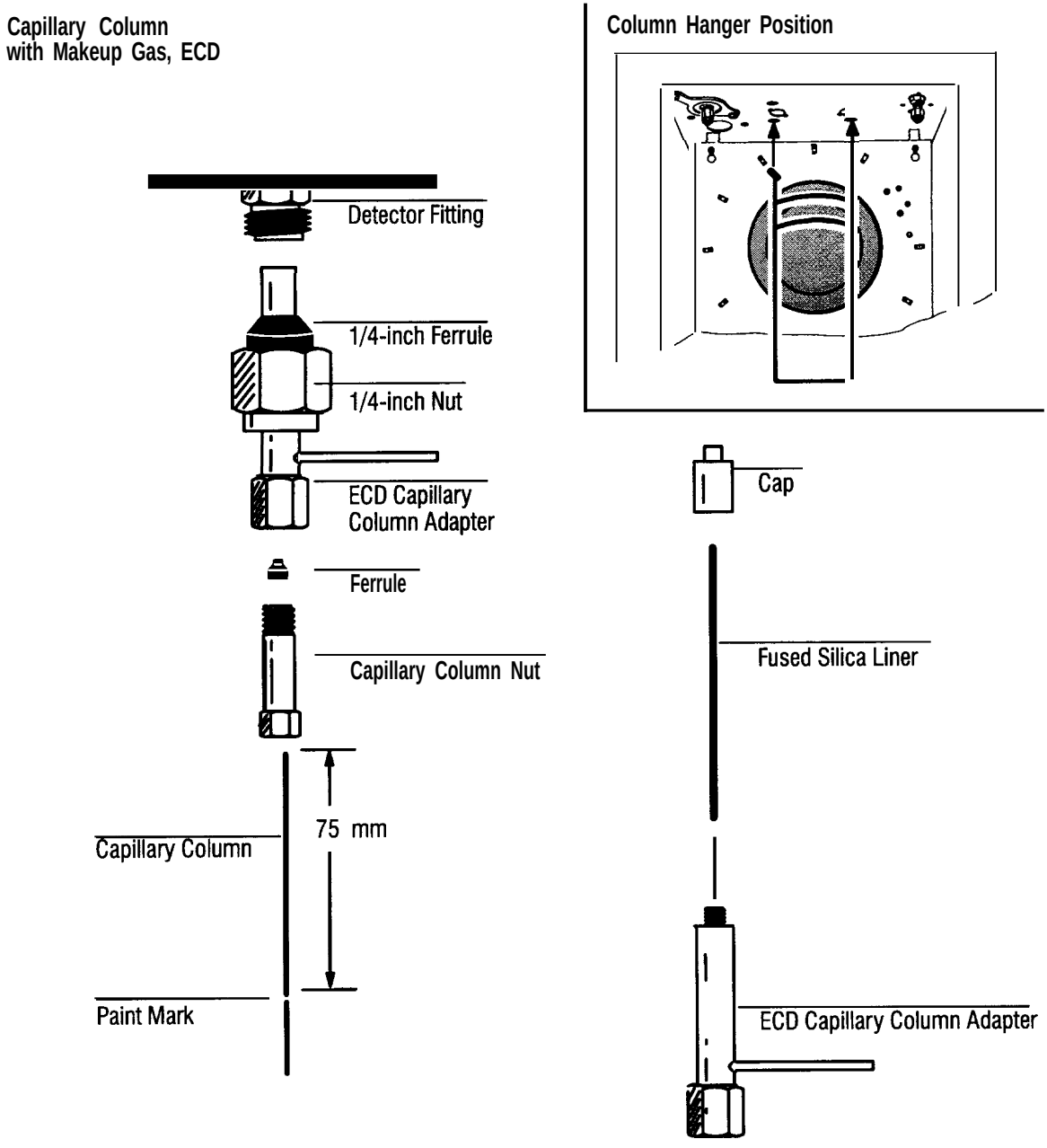
Installing a l/4-inch Glass Column in an Electron Capture Detector

Installing a capillary column in an electron capture detector

Using the figures on the next page as a guide:

1. Remove the cap of the makeup gas adapter.
2. Install a fused silica liner in the bottom half of the adapter.
3. Replace the cap of the makeup gas adapter. Tighten the cap hand-tight.
4. Insert the ECD adapter straight into the detector as far as possible and tighten the nut finger-tight.
5. Use a wrench to tighten the nut an additional l/4-turn.
6. Install on the column a column nut and ferrule. Note that either a 1. O- or 0.5-mm id graphite ferrule may be used depending upon column outer diameter.

   Inserting the column through the nut and ferrule may contaminate the end of the column. Prepare a fresh column end following instructions given in *Preparing fused silica capillary columns* in this chapter.
7. Measure 75 mm from the end of the column and mark the column. Typewriter correction fluid is a good marking material. Gently insert the column into the detector followed by the ferrule and column nut. Tighten the nut finger-tight. Position the column so the 75-mm mark is even with the end of the column nut. Tighten the nut an additional l/4-turn with a wrench.



Installing a Capillary Column in an Electron Capture Detector

Installing an l/8-inch metal column in a flame photometric detector

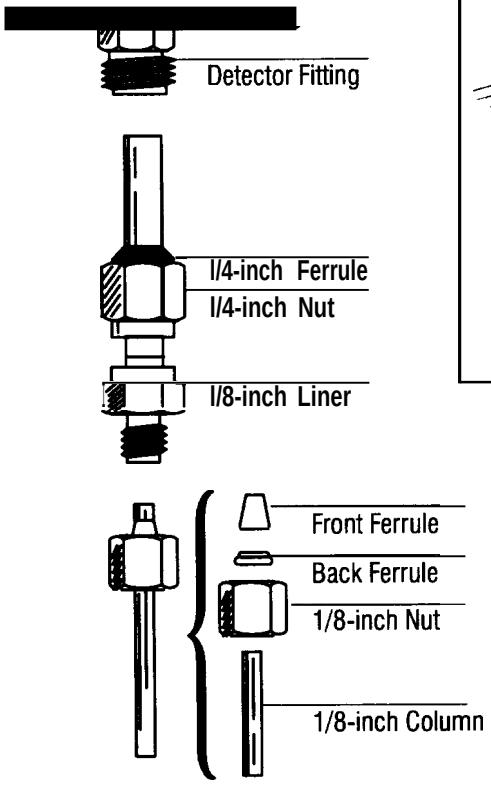
To install new swage-type nut and ferrules on the column, follow the procedure *Preparing packed metal columns*, earlier in this chapter. For columns with ferrules already installed, continue.

Using the figures on the next page as a guide:

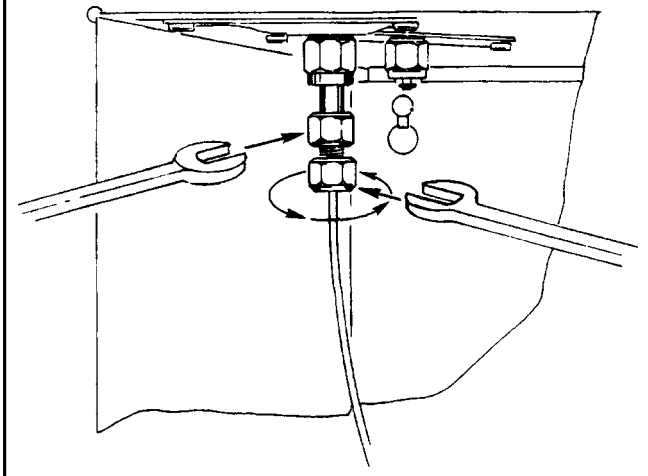
1. Assemble a brass nut and graphite ferrule onto the liner/adapter.
2. Insert the adapter straight into the detector base as far as possible.
3. Holding the adapter in this position, tighten the nut finger-tight.
4. Use a wrench to tighten the nut an additional l/4-turn.
5. Install the column into the inlet by tightening the column nut assuming ferrule(s) are already set (locked) onto the column (see *Preparing packed metal columns* in this chapter). An additional l/4-turn is usually sufficient.

   Use two wrenches in opposition, one on the column nut and the other on the liner body, to prevent rotation of the liner while tightening the column nut.

l/8-inch Packed Metal Column, FPD



Using Two Wrenches in Opposition to Tighten Column Fittings



Installing an l/8-inch Metal Column in a Flame Photometric Detector

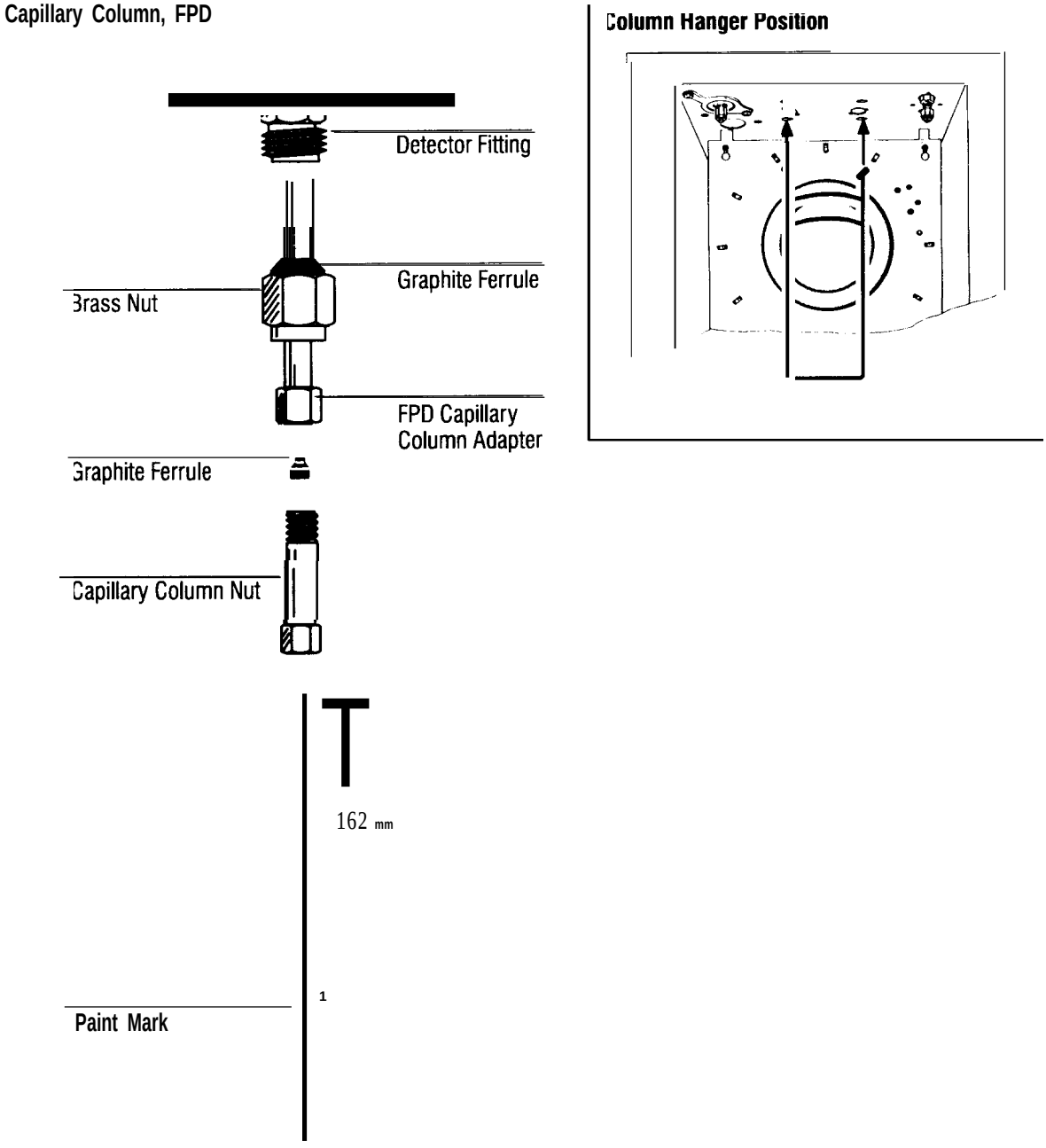
Installing a capillary column in a flame photometric detector

Using the figures on the next page as a guide:

1. Assemble a brass nut and graphite ferrule onto the FPD capillary column adapter.
2. Insert the FPD adapter straight into the detector as far as possible and tighten the nut finger-tight.
3. Use a wrench to tighten the nut an additional l/4-turn.
4. Install on the column a column nut and ferrule. Note that either a 1. O- or 0.5-mm id graphite ferrule may be used depending upon column outer diameter.

   Inserting the column through the nut and ferrule may contaminate the end of the column. Prepare a fresh column end following instructions given in *Preparing fused silica capillary columns* in this chapter.
5. Measure 162 mm from the end of the column and mark the column. Typewriter correction fluid is a good marking material. Gently insert the column into the detector followed by the ferrule and column nut. Tighten the nut finger-tight. Position the column so the 162-mm mark is even with the end of the column nut. Tighten the nut an additional l/4-turn with a wrench.

   This height may be optimized higher or lower depending on sample type and detector flow rates. If the column is too high, it can be exposed to the detector flame. If the column is too low, the sample can be exposed to some hot stainless steel which can result in slight peak tailing.

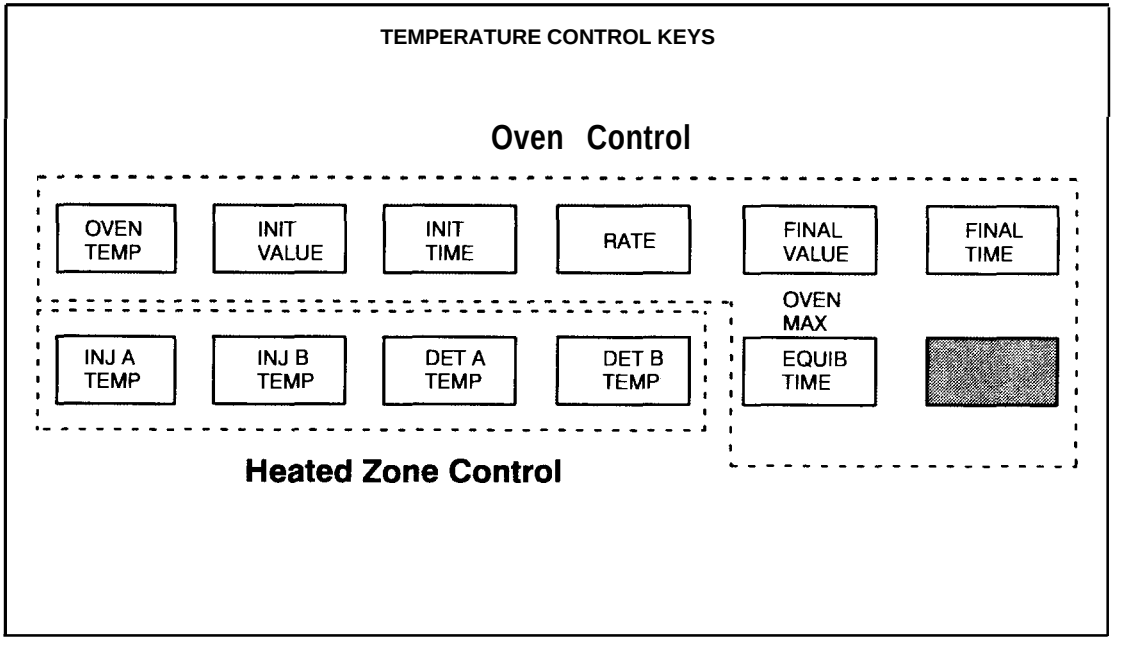


Installing a Capillary Column in a Flame Photometric Detector

Setting Heated Zone Temperatures

Setting Heated Zone Temperatures

Oven temperature, and temperatures of up to five separate heated zones (detectors, inlets, and/or heated valves), are controlled through keys shown.



In these cases, both current setpoint value and current monitored value are displayed by pressing the appropriate temperature control key. For example, the next figure shows typical displays obtained by pressing the OVEN TEMP key.

	ACTUAL	SETPOINT
OVEN TEMP	279	350

Typical Display, Setpoint And Current Value

Note that the **ACTUAL** value is a measured quantity, while the **SETPOINT** value is user-defined: In this example, the setpoint value for oven temperature might recently have been changed from 250 to 350° C, and the oven is now heating to the new setpoint. Given sufficient time for equilibration, **ACTUAL** and **SETPOINT** values become equal.

In addition to keys [0] through [9], [−], [•], [CLEAR], and [ENTER], which are used in defining setpoint values, [ON], [OFF], [A], and [B] are used in certain specific key sequences:

- Keys [ON] and [OFF] add convenience in being able to switch on or off the oven, and/or heated zones, without losing their current setpoint values.
- Keys [A] and [B] are used in key sequences defining a multiple-ramp oven temperature program: [A] as part of key sequences defining parameters for the second ramp, [B] as part of key sequences defining parameters for the third ramp.

Operating limits for heated zones

Valid Setpoint Ranges For Temperature Control Keys

Key	Valid Setpoint Range	In Increments of	Function
OVEN TEMP	–80 to 450	1“c	Oven Control
INIT TEMP	–80 to 450	1“c	Oven Control
INIT TIME	0 to 650.00	0.01 minute	Oven Control
RATE	0 to 70	0.1 /minute	Oven Control
FINAL TEMP	–80 to 450	1“c	Oven Control
FINAL TIME	0 to 650.00	0.01 minute	Oven Control
OVEN MAX	70 to 450	1“c	Oven Control
EQUIB TIME	0 to 200.00	0.01 minute	Oven Control
INJ A TEMP	0 to 400	1“c	Zone Control
INJ B TEMP	0 to 400	1“c	Zone Control
DET A TEMP	0 to 400’	1“c	Zone Control
DET B TEMP	o to 400’	1“c	Zone Control
AUX TEMP	o to 400	1“c	Zone Control

NOTE: TOTAL run time will not exceed 650.00 minutes regardless of values entered for INIT TIME, RATE and FINAL TIME

*The valid setpoint range for a flame ionization detector is O to 450°C.

Setting oven temperatures

Oven temperature may be controlled anywhere within the range of –80°C (with cryogenic cooling using liquid N_2) through 450°C in increments of 1°C.

Oven temperature control keys include:

OVEN TEMP	To enter a constant temperature for the oven
EQUIB TIME	To enter a time for oven temperature to equilibrate whenever oven temperature is modified *(Equilibration time begins when the actual oven temperature comes within 1°C of the oven temperature setting.)*
OVEN MAX	To set an oven temperature maximum limit

Displaying oven temperature

Press OVEN TEMP to display the current oven temperature.

Sample Display = **OVEN TEMP 50 50 (or OFF)**

The oven is switched on with key sequence: OVEN TEMP ON ENTER

The oven is also switched on by entering a new setpoint value; the new value replaces OFF or the previous value.

Examples

Setting oven temperature to 200°C

Current oven temp = 100°C

Display = **OVEN TEMP 100 100**

Press: OVEN TEMP 2 0 0 ENTER

The oven temperature will change from 100°C to 200°C and stabilize.

Setting oven equilibration time to 1 minute

Press: EQUIB TIME 1 . 0 0 ENTER

The oven equilibration time will be 1 minute.

Setting the oven maximum to 350°C

Press: gold OVEN MAX 3 5 0 ENTER

The maximum temperature the oven can be set to is 350°C.

The HP 5890 SERIES II verifies oven setpoints as they are entered; an appropriate message is displayed when an entered setpoint is inconsistent with a previously defined setpoint.

Display = **OVEN MAXIMUM = 350**

In this case, an oven temperature greater then 350°C was attempted while the **OVEN MAX is set to 350°C.**

Using cryogenic oven cooling

Oven temperature may be controlled below ambient when a cryogenic valve is present.

Cryogenic control setpoints are:

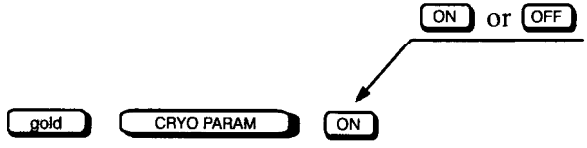
CRYO ON	To enable subambient control of the oven
CRYO OFF	To disable subambient cooling of the oven; the default state for the cryogenic valve is OFF.
CRYO BLAST ON	To enable very fast cool-downtime after a run
CRYO BLAST OFF	To disable very fast cooling of the oven
AMBIENT	Sets optimal temperature control 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 WARN:OVEN SHUT OFF is displayed. Turning Cryo Fault OFF will disable this feature.
CRYO TIMEOUT XXX MIN	A cryo timeout occurs when a run does not start within a specified time (1 O to 120 minutes) after the oven equilibrates. Turning Cryo Timeout OFF will disable this feature. Default is ON for 30 minutes.

When on, the cryogenic valve (if installed) operates automatically to obtain an oven temperature when there is demand for coolant to be supplied to the oven.

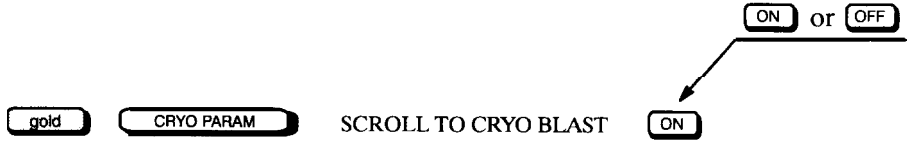
When cryogenic cooling is not needed, cryogenic valve operation must be turned off. If this is not done, proper oven temperature control may not be possible, particularly at temperatures near ambient.

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 normally would under normal cryogenic operation. This allows the HP 5890 to become 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 functions related to cryogenic valve operation. To turn cryogenic operation on/off and cryogenic blast on/off, the following key sequence is used:



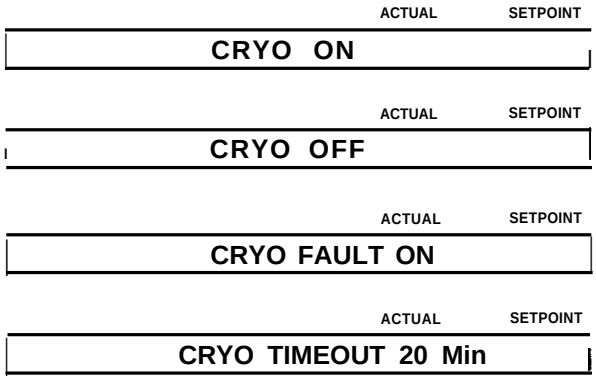
To turn Cryo Blast operation on or off, the following key sequence is used:



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

Ambient temperature is setable to allow fine tuning of cryogenic operation. The default setting is 25°C and for most applications need not be changed. For more information about adjusting the ambient cryogenic setting, see the *HP 5890 SERIES II Reference Manual.*

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



Displays, Cryogenic Valve Operation

Programming oven temperatures

The oven temperature may be programmed from an initial temperature to a final temperature (in any combination of heating or cooling) using up to three ramps during a run.

Oven temperature programming keys include:

INIT VALUE	**The starting temperature of a temperature programmed run. This is also the temperature the oven returns to at the end of a temperature programmed run.**
INIT TIME	**The length of time the oven will stay at the starting temperature after a programmed run has begun.**
RATE	**Controls the rate at which the oven will be heated or cooled in** degrees C/min. *A tempeture-programming rate of 0 halts* further programming.
FINAL VALUE	**Temperature the oven will reach at the end of a heating or cooling temperature-programmed run.**
FINAL TIME	**The length of time the oven temperature will be held at the final temperature of a temperature-programmed run.**

Total elapsed time for a run cannot exceed 650 minutes. At 650 minutes, the run terminates and oven temperature returns to the initial oven temperature. In isothermal operation **(RATE = O),** the instrument internally sets run time to the maximum of 650 minutes.

Examples

A single-ramp temperature program

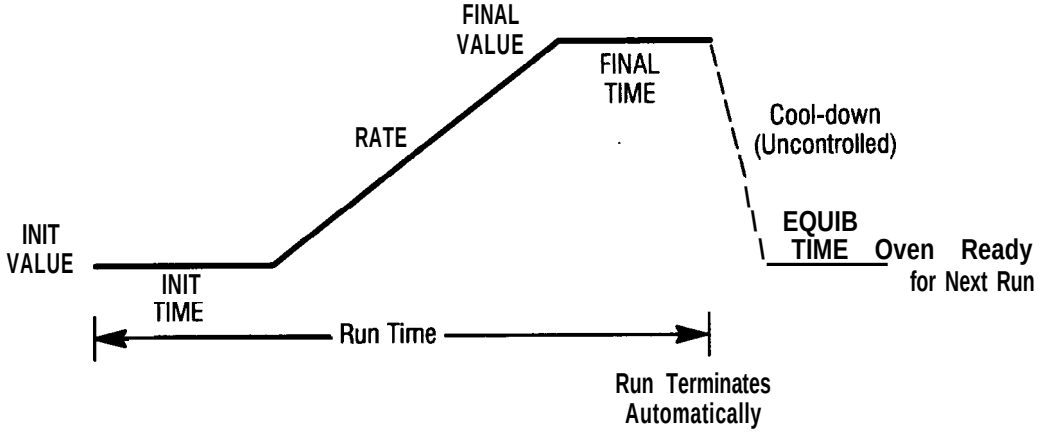
Programming oven temperature from 100°C to 200°C at I0°C/min.

Current oven temp = 100°C,

Display = | OVEN TEMP | 100 | 100 |

INIT VALUE	100
INIT TIME	2
RATE	10
FINAL VALUE	200
FINAL TIME	1

Single-Ramp Temperature Program



Example setpoints for a single-ramp temperature program

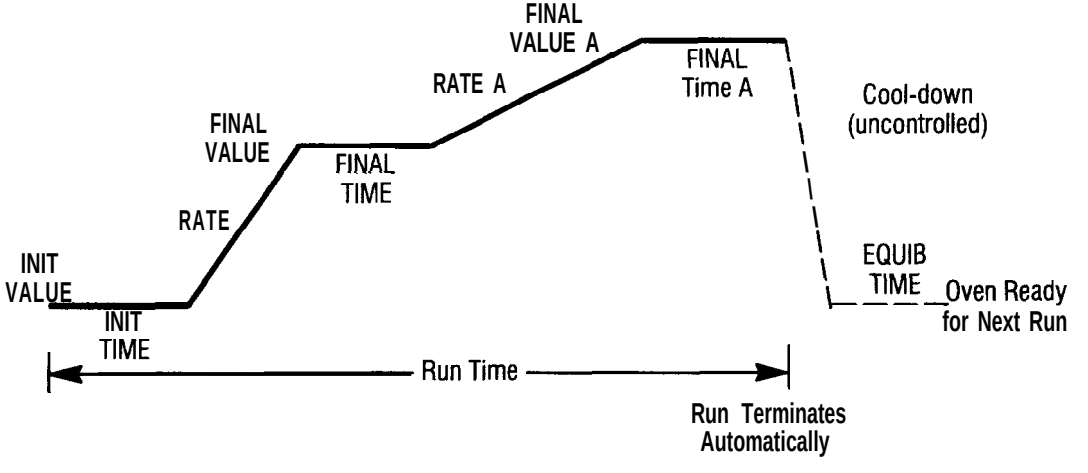
A two-ramp temperature program

Oven temperature will be held at 100° C for 2 minutes, then program from 100°C to 200°C at I0° C/min for the first ramp.

Oven temperature will be held at 200° C for 2 minutes, then program from 200°C to 250°C at 5°C/min for the second ramp.

	1ST RAMP	2ND RAMP	
INIT VALUE	100		
INIT TIME	2		
RATE	10	RATE A	5
FINAL VALUE	200	FINAL VALUE	250
FINAL TIME	2	FINAL TIME	1

Two-Ramp Temperature Program



Example setpoints for a two ramp oven program

A three-ramp temperature program

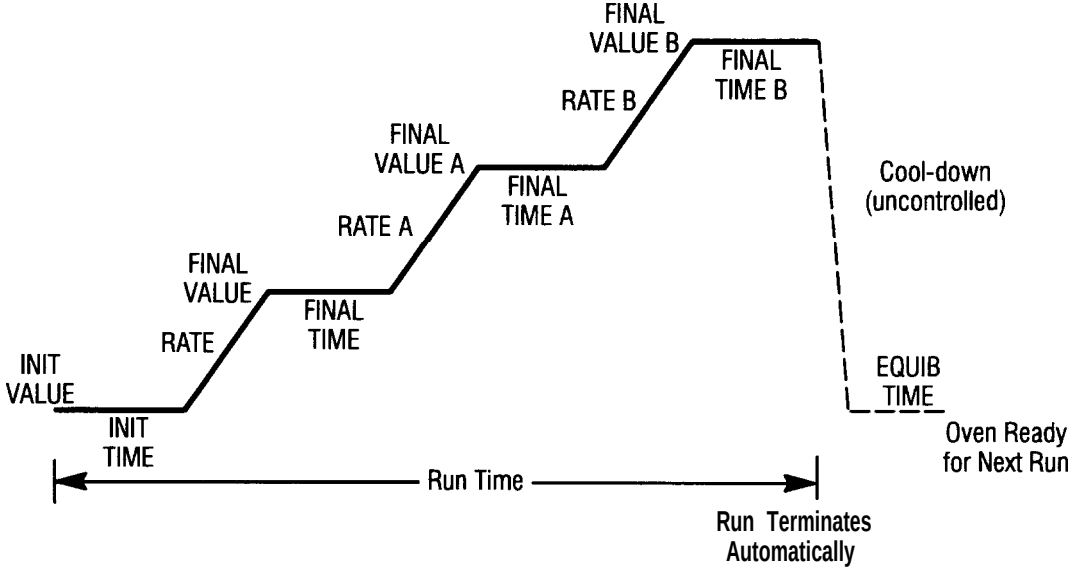
Oven temperature will be held at 100” C for 1 minute, then program from 100^{0}C to 200^{0}C at 10°C/min for the first ramp.

Oven temperature will be held at 200^{0}C for 2 minutes, then program from 200^{0}C to 250°C at 10°C/min for the second ramp.

Oven temperature will be held at 250° C for 2 minutes, then program from 250^{0}C to 300^{0}C at 10°C/min for the third ramp. Oven temperature will be held at 300°C for 1 minute before returning to the initial starting temperature of 100^{0}C.

1ST RAMP		2ND RAMP		3RD RAMP	
INIT VALUE	100				
INIIT TIME	1				
RATE	10	**RATE A**	10	**RATE B**	10
FINAL VALUE	**200**	FINAL VALUE	**250**	FINAL VALUE	**300**
FINAL TIME	**2**	FINAL TIME	**2**	FINAL TIME	1

Three-Ramp
Temperature Program



Example setpoints for a three-ramp oven program

A three-ramp temperature program (with a controlled cool-down step)

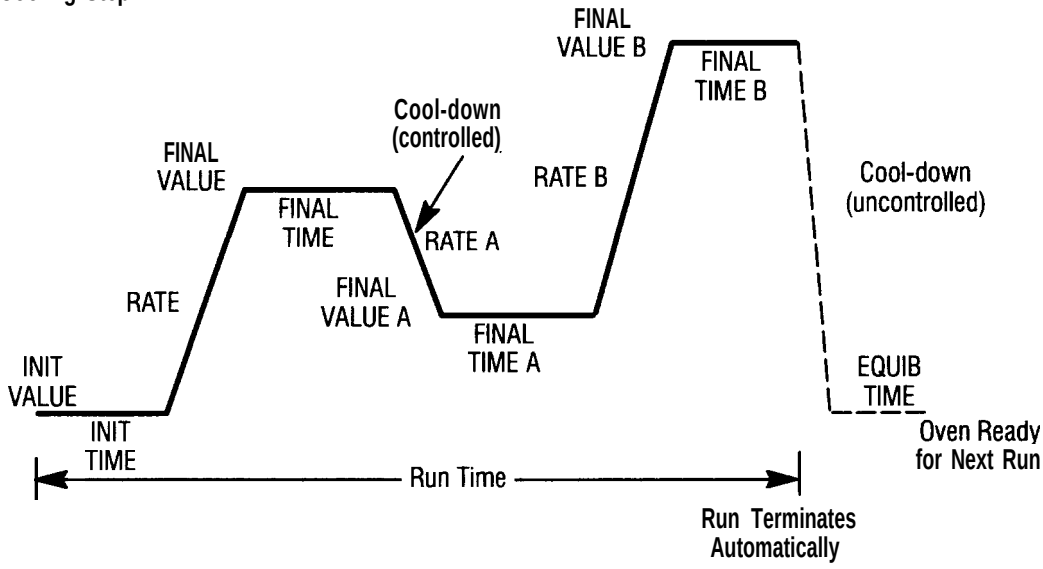
Oven temperature will be held at 100°C for 1 minute, then program from 100°C to 200°C at 10° C/min for the first ramp.

Oven temperature will be held at 200°C for 2 minutes, then cool (controlled) from 200°C to 150°C for the second ramp.

Oven temperature will be held at 150°C for 1 minute, then program from 150°C to 250°C at I0°C/min for the third ramp. Oven temperature will be held at 250°C for 2 minutes before returning to the initial starting temperature of 100°C.

1ST RAMP		2ND RAMP		3RD RAMP	
INIT VALUE	100				
INIT TIME	1				
RATE	10	RATE A	5	RATE B	10
FINAL VALUE	200	FINAL VALUE	150	FINAL VALUE	250
FINAL TIME	2	FINAL TIME	1	FINAL TIME	2

Three-Ramp Temperature Program with a Cooling Step



Example setpoint conditions for a three-ramp oven program

Setting inlet and detector temperatures

Inlet and detector temperatures may be controlled anywhere from room temperature to 400° C.

Inlet and detector temperature control keys include:

INJ A TEMP	To set temperature for the inlet in the A position
INJ B TEMP	To set temperature for the inlet in the B position
DET A TEMP	To set temperature for the detector in the A position
DET B TEMP	To set temperature for the detector in the B position

Displaying inlet and detector temperature

Press [INJ A TEMP] (or [INJ B TEMP]) to display the current inlet temperature.

Sample Display = | INJ A TEMP 100 100 |

Press [DET A TEMP] (or [DET B TEMP]) to display the current detector temperature.

Sample Display = | DET A TEMP 100 100 |

An inlet or detector is switched on or off with key sequences:

([INJ A TEMP] or [INJ B TEMP]) [ON] / [OFF]

([DET A TEMP] or [DET B TEMP]) [ON] / [OFF]

The inlet or detector is also switched on by entering a new setpoint value; the new value replaces **OFF** or the previous value.

Example

Setting inlet temperature to 200°C

Current inlet temp = **OFF**

Display = | **INJ A TEMP** 38 **OFF** |

Press: (INJ A TEMP or INJ B TEMP) 2 0 0 ENTER

The inlet temperature will change from OFF to 200°C and stabilize.

Setting detector temperature to 200°C

Current detector temp = **OFF**

Display = | **DET A TEMP** 38 **OFF** |

Press: (DET A TEMP or DET B TEMP) 2 0 0 ENTER

The detector temperature will change from OFF to 200°C and stabilize.

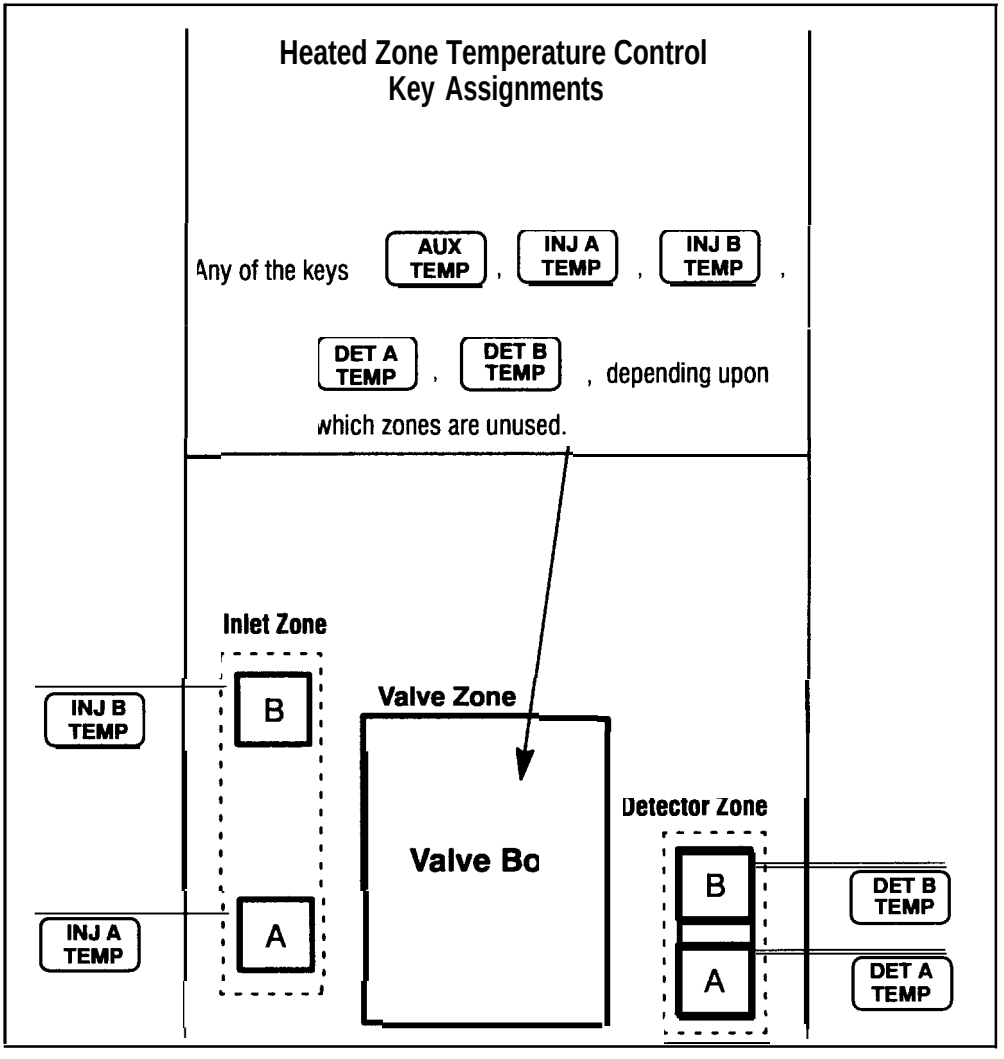
Setting auxiliary temperatures

AUX TEMP	AUX TEMP is usually used for heated valve compartment, heated transfer line, etc.
INJ A TEMP	Any of these keys may control an auxiliary heated zone depending upon the instrument configuration.
INJ B TEMP	
DET A TEMP	
DET B TEMP	

Instructions are provided in a separate envelope when a temperature control key has been assigned to a heated zone other than the zone it identifies.

As an example, to **set** the **AUX TEMP** heated zone to 100°C,

Press: gold AUX TEMP 1 0 0 ENTER



Instrument Rear

4

Setting Inlet System Flow Rates

Setting Inlet System Flow Rates

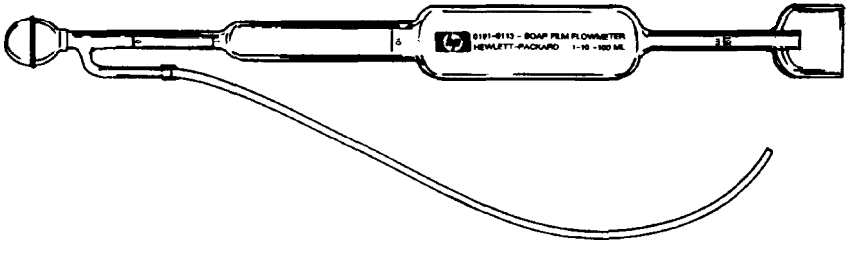
This chapter provides operating information for the following HP 5890 inlet systems:

- Septum-purged packed column inlet
- Split/splitless capillary inlet

Operating information for the Programmable Cool On-Column Inlet is provided in a separate manual included with the HP 5890.

Measuring flow rates

Use a bubble flow meter to initialize all flows for the first time and to check them whenever the system is changed in some way.



Bubble Flow Meter for Measuring Flow Rates

Using a bubble flow meter

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).

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.

1. Attach one end of the bubble flow meter adapter to the flexible gas-inlet line of the bubble flow meter.
2. Attach the other end of the adapter to the detector outlet exhaust vent or other vent through which you will measure flow.
3. Fill the bulb of the bubble flow meter with soapy water or leak detection fluid (such as Snoop®).
4. Prepare the built-in stopwatch on the keyboard using the following keystrokes on its keypad:

   Press: TIME up to three times.

   The display on the oven module now displays zeroes for the time (t) and the reciprocal time (l/t).
5. While holding the bubble flow meter vertically squeeze and release the bulb to create a meniscus in the bubble flow meter.
6. Press: ENTER to start the stopwatch when the meniscus passes the lowest line in the bubble flow meter.
7. Press: ENTER to stop the stopwatch when the meniscus passes the upper line in one of the tube sections.
8. Calculate the flow rate in ml/min:

   . If you stopped the meniscus at the first line, the flow rate is numerically equivalent to the reciprocal time reading displayed on the oven module.

   ● If you stopped the meniscus at the second line, the flow rate is numerically equivalent to 10 times the reciprocal time reading displayed on the oven module.

   ● If you stopped the meniscus at the third line, the flow rate is numerically equivalent to 100 times the reciprocal time reading displayed on the oven module.
9. Press: CLEAR and repeat steps 5 through 8 at least once to verify the flow.
10. Start the makeup gas flow by turning the Aux Gas knob on the upper-left portion of the oven front counterclockwise until the valve is in the open position.

11. Measure the total gas flow by repeating steps 5 through 8.
12. If the flow is not correct:
    - Wait at least 2 minutes for the flow through the system to stabilize.
    - Repeat the above procedure as necessary.

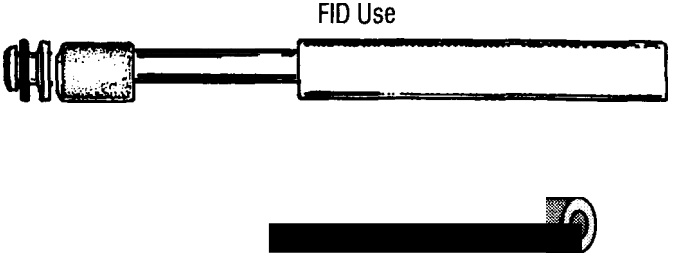
    Note: If you use an FID, TCD, or NPD, use a small screwdriver to adjust the variable restrictor at the center of the Aux Gas knob as necessary.

Required adapters for measuring flow rates

In general, inlet system, or column, flow rates are measured at detector exhaust vents. Septum purge and split flow rates for capillary inlet systems are measured at vents located on the front of the flow panel. A rubber adapter tube attaches directly to an NPD, ECD, or TCD exhaust vent tube.

A special flow-measuring adapter is supplied for an FID. Attach a bubble flow meter to the FID flow-measuring adapter and insert it into the detector exhaust vent as far as possible. You may feel initial resistance as the adapter's O-ring is forced into the detector exhaust vent. Twist and push the adapter during insertion to ensure that the O-ring forms a good seal.

WARNING To minimize the risk of explosion, never measure air and H_2 together. Measure them separately.



Required Adapters for Measuring Gas Flow Rates

Changing the packed inlet flow ranges

You may want to change the flow range of your inlet for a number of reasons. For example, if you are using flows in the lowest 20 percent of a flow restrictor's range, the retention times of your analysis might wander. By changing from a flow of 20 ml/min with a flow restriction range of O to 110 ml/min to one with a range of O to 20 ml/min, you can eliminate this problem.

You can change the flow ranges in packed inlets by either:

- Changing the source pressure, or
- Changing the flow restrictor in the flow controller. For instructions on changing the flow restrictor, see the section on *Changing the flow restrictor* in the *Site Prep/Installation Manual.*

Changing the source pressure

You can increase the upper limit of flow from a flow controller by increasing the source pressure. The following table lists the maximum flows for the standard flow controller for a packed inlet with a O to 20 ml/min flow restrictor at five pressures. For maximum H_2 flows, read from the Helium Flow column.

Source Pressure (psi)	Nitrogen Flow (ml/min)	Helium Flow (ml/min)
40	20	21
50	24	25
60	28	28
70	32	32
80	36	35

Setting the packed inlet flow with septum purge

Use the following steps to set the packed inlet flow:

1. Set the oven and heated zone temperatures to the desired operating values.

   Note: Never heat the column until the flow rates are set.

2. Turn off the detector (particularly an NPD or TCD), if it is not off already until you set the carrier flow rate.

   Note: The detector signal can be assigned to an appropriate output channel.

3. Set the carrier source pressure to at least 275 kPa (40 psi) to ensure proper operation for most applications.

   Note: Carrier source pressure must beat least 105 kPa (15 psi) greater than the maximum column head pressure.

4. Turn off any other support gases to the detector (such as H_2, air, reference flow, or capillary makeup gas) to permit independent measurement of column flow rate.

5. Your inlet is equipped with either manual or electronic flow control. Set the column head pressure according to the appropriate section below.

   Manual flow control:

   Turn the mass flow controller counterclockwise as necessary to obtain the desired flow rate, as measured with a bubble flow meter at the detector exhaust vent.

Electronic pressure control:

a. Select the pressure units you would like to use.

To change the units, press: gold 1 ENTER . Then press the number of the corresponding unit you want to use:

1 = psi
2 = bar
3 = kPa

b. The example below sets Inlet B (Injector B) pressure to 10 psi. Use the example to set the pressure you have selected.

c. Press:

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

	ACTUAL	SETPOINT
EPP B	10.0	10.0

The GC display looks like this

Note: To keep the pressure constant through an oven ramp program, see Chapter 10, *Using Electronic Pressure Control.*

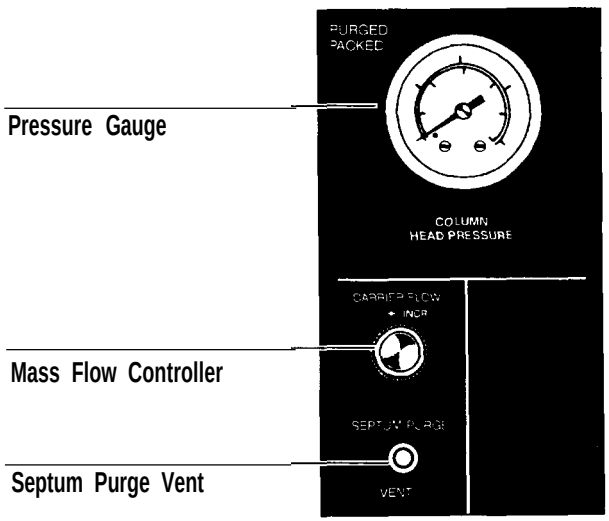
6. Check the septum purge flow rate:

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

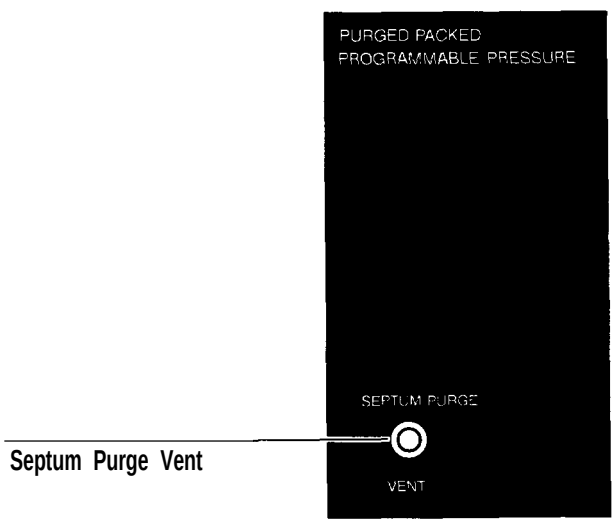
Carrier Gas Type	Approx. Flow
H_2	1.2–2.2 ml/min
He	1.0–1.8
N_2	0.6–1.2
Argon/Methane	0.5–1.1

7. Recheck the column flow rate and adjust as necessary.

Manual Pressure Control



Electronic Pressure Control



Flow Panels Controlling Purged Packed Inlet

Setting the split/splitless capillary inlet flow

Set the linear velocity through the column when using capillary columns. Linear velocity is controlled by pressure at the head of the column. Pressure required to obtain a particular velocity depends primarily upon the bore (id) of the column, length of the column, and oven temperature.

Hewlett-Packard fused-silica capillary columns are categorized according to their bores. The table below lists the initial pressures for some capillary column bores and lengths.

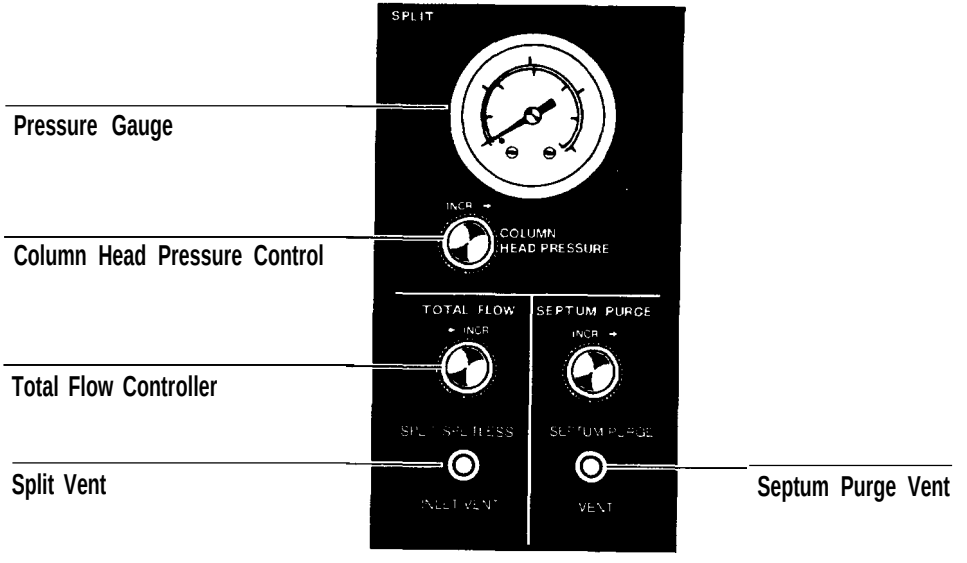
The high pressure in each range is recommended as a starting point for most analyses and yields a good compromise between efficiency and speed of analysis. The following sections provide procedures to adjust head pressure to obtain any desired flow velocity through the column.

Suggested Initial Head Pressures for Capillary Columns

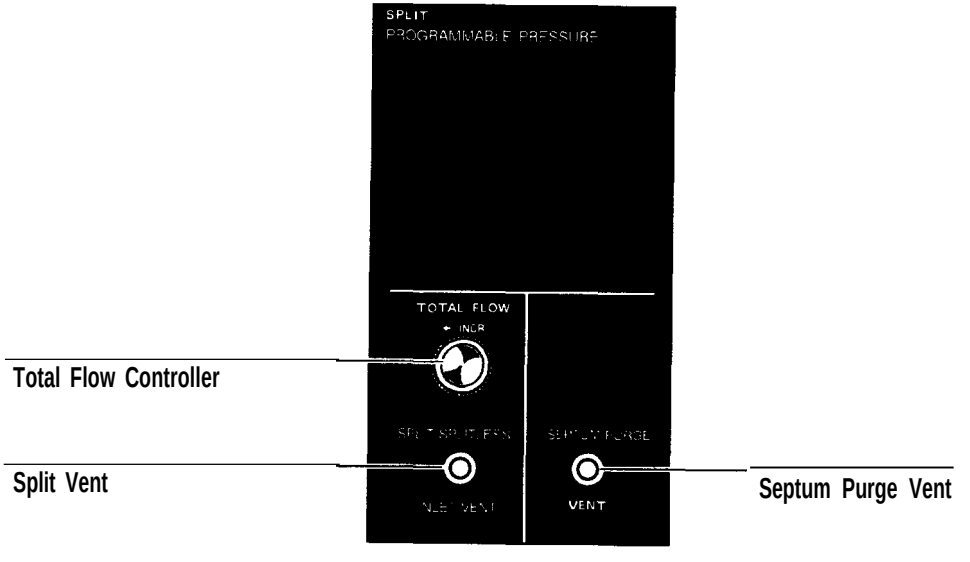
Column id (mm)	Column Length (m)	Helium Carrier Gas kPa	Helium Carrier Gas psi	Hydrogen Carrier Gas kPa	Hydrogen Carrier Gas psi
0.20	12	85 - 140	12 - 21	48 - 84	7 - 12
0.20	25	145 - 235	21 - 34	87 - 145	13 - 21
0.20	50	235 - 360	34 - 52	145 - 230	21 - 34
0.32	12	29 - 53	4.2 - 7.7	17 - 32	2.5 - 4.7
0.32	25	55 - 95	7.9 - 14	33 - 60	4.8 - 8.7
0.32	50	95 - 160	14.0 - 23	60 - 105	8.7 - 15
0.53	10	8.5 - 16	1.2 - 2.4	5.0 - 9.7	0.7 - 1.4
0.53	30	24.0 - 44	3.5 - 6.3	14.0 - 27	2.1 - 3.9

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

Manual Pressure Control



Electronic Pressure Control



Flow Panels Controlling Split/Splitless Inlet

Setting the split mode flow

WARNING When performing split sampling and using hazardous chemicals and/or H_2 carrier gas, vent effluent from the split vent and septum purge vent to a fume hood or appropriate chemical trap.

To ensure proper operation, make sure the carrier source pressure is at least 105 kPa (15 psi) greater than the selected column head pressure.

1. Use the following steps to set the initial column head pressure:

   a. Set the column head pressure to O:

   Press: gold INJ A PRES (or INJ B PRES) 0 ENTER .

   b. Increase the total flow control as necessary to obtain 100 ml/min measured at the split vent.

   c. Increase the column head pressure to obtain the selected pressure.

   Your inlet is equipped with either manual or electronic pressure control. Set the column head pressure according to the appropriate instructions below.

   d. Set the oven and heated zone temperatures to the desired operating values. Make sure the detector is turned on and its output signal is assigned to an appropriate channel (see Chapter 6, *Controlling the Signal Output).*

   Manual flow control:

   Turn the mass flow controller counterclockwise to establish flow. This will cause the gauge pressure to increase.

   Electronic pressure control:

   a. Select the pressure units you would like to use.

   To change the units, press: gold 1 ENTER **. Then press the** number of the corresponding unit you want to use:

   1 = psi
   2 = bar
   3 = kPa

b. The example below sets Inlet B (Injector B) pressure to 10 psi. Use the example to set the pressure you have selected.

c. Press:

gold | INJ B PRES | 1 | 0 | ENTER — *Sets inlet B pressure to 10 psi*

	ACTUAL	SETPOINT
EPP B	10.0	10.0

The GC display looks like this

Note: To keep the pressure constant through an oven ramp program, see Chapter 10, *Using Electronic Pressure Control.*

2. Check the septum purge flow rate.

Excess carrier gas is vented through the septum purge vent. Although the septum purge vent is not adjustable, you should check the flow. Do not cap off the flow from the purge vent.

Carrier Gas Type	Approx. Flow
H_2	3.5-6.0 ml/min
He	1.5-3.5 ml/min
N_2	1.5-3.5 ml/min
Argon/Methane	1.5-3.5 ml/min

3. Set the linear velocity:

Using the timer feature (see *Using the Internal Stopwatch* in this chapter) and repeated injection of an unretained component, adjust the column head pressure as necessary to obtain the expected retention time for the desired linear velocity

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

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

$$t_{r\ (expected)}\ (\text{in min}) = 1.67 \frac{\text{Column Length (m)}}{\text{Linear Flow Velocity (cm/sec)}}$$

4. Calculate the volumetric flow rate, if desired:

   Use the following formula to simplify the calculation of volumetric flow through a capillary column:

$$\text{Volumetric Flow Rate } (cm^3/min) = 0.785 \frac{D^2 L}{t_r}$$

   where D is column internal diameter
   L is column length
   t_r is retention time (min) of an unretained component, assuming the desired linear velocity (p.) has been obtained

   This calculation becomes less accurate as pressure and gas compression are increased.

   The table below lists values of $0.785 \times D^2 L$ for capillary column bores and lengths:

Values of $0.785 \times D^2 L$ for Various Capillary Column Bores and Lengths

Nominal id (mm)	Nominal Length (m)		
	12	25	50
0.20	0.377	0.785	1.57
0.25	0.589	1.22	2.45
0.32	0.965	2.01	4.02
0.53	2.65	5.51	11.0
0.75	5.30	11.0	22.1

5. Use a bubble flow meter connected at the detector exhaust vent to verify the calculated volumetric flow rate through the column. (For bubble flow meter operating instructions, see *Using a bubble flow meter* earlier in this chapter.) Turn off any other gases to the detector, such as makeup and/or support gases.

6. Use the following steps to verify that the inlet split flow is currently passing through the inlet insert and will remain so throughout runs that are made in split sampling mode:

   a. Display the current inlet split vent status:

      Press: PURGE/VALVE A (or B).

      If OFF is displayed, press: ON to restore split flow through the inlet insert.

   b. Display the time at which the split flow will be halted:

      Press: PURGE/VALVE A (or B) TIME OFF.

   c. Display the time at which the split flow will be restored:

      Press: PURGE/VALVE A (or B) TIME ON.

      (or just press: ON if VALVE TIME is already displayed).

   d. Alternatively, set both times to 0.00 and turn on Purge A (or B):

      Press: 0 ENTER when the GC display reads:

7. Use the following steps to obtain the desired split ratio:

   a. Measure the flow rate at the split vent using a bubble flow meter. For bubble flow meter operating instructions, see *Using a bubble flow meter* in this chapter

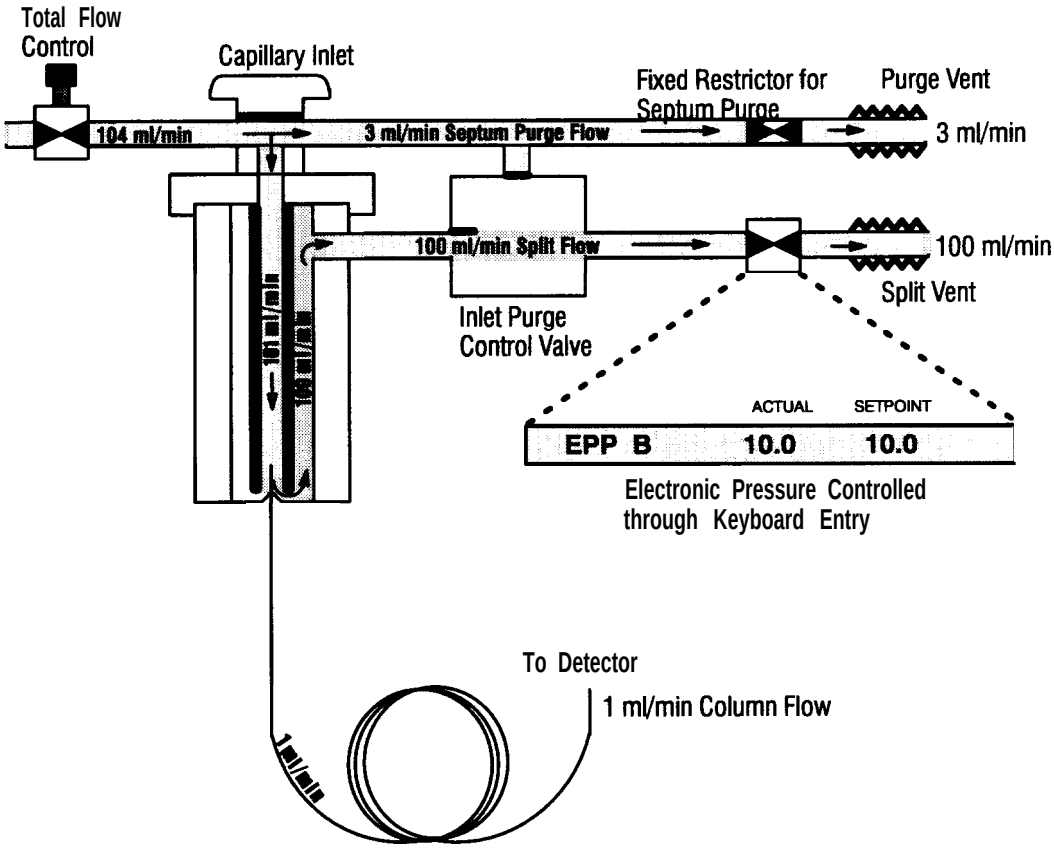
   b. Adjust the total flow control as necessary to obtain the flow rate required for the desired split ratio.

   c. Choose the split ratio appropriate for the analysis.

      From the definition for the split ratio, use the following relationship to determine the flow rate to be expected at the split vent for any desired split ratio:

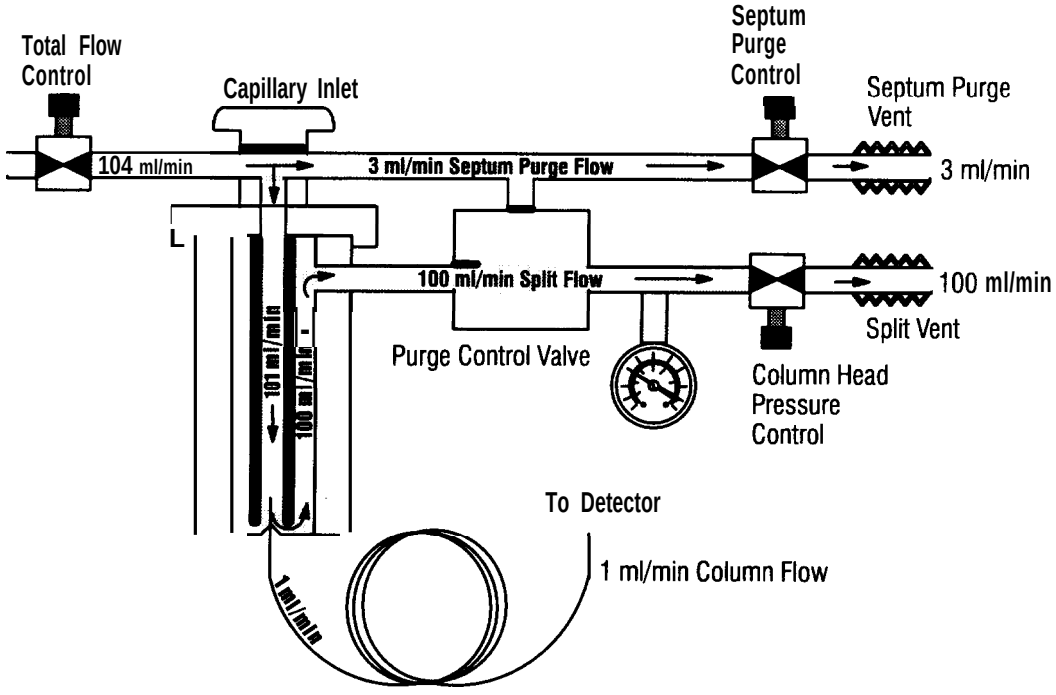
      Split Vent Flow Rate (ml/min) =

      Volumetric Column Flow Rate (ml/min) x (Desired Split Ratio - 1)



This example shows a 100:1 split ratio
100 ml/min Split Flow
1 ml/min Column Flow

Split Flow Diagram for Electronic Pressure Control



This example shows a 100:1 split ratio
100 ml/min Split Flow
1 ml/min Column Flow

Split Flow Diagram for Manual Pressure Control

Setting the splitless mode flow

WARNING When performing splitless sampling and using hazardous chemicals and/or H_2 carrier gas, vent effluent from the split vent and septum purge vent to a fume hood or appropriate chemical trap.

To ensure proper operation, make sure the carrier source pressure is at least 105 kPa (15 psi) greater than the selected column head pressure.

Use these steps to set the splitless mode flow. This procedure assumes that detector gases are connected, the system is leak-free, and the column and insert are properly installed.

1. Use the following steps to set the initial column head pressure:

   a. Set the column head pressure and total flow controls to O.

   Press: [gold] [INJ A PRES] (or [INJ B PRES]) [0] [ENTER]

   Sets *the EPC inlets to O*

   b. Increase the total flow control as necessary to obtain 50 ml/min measured at the inlet vent.

   c. Increase the column head pressure to obtain the selected pressure.

   Your inlet is equipped with either manual or electronic pressure control. Set the column head pressure according to the following instructions for your inlet.

   d. Set the oven and heated zone temperatures to the desired operating values. Make sure the detector is turned on and its output signal is assigned to an appropriate channel (see Chapter 6, *Controlling Signal Output).*

Manual flow control:

Turn the mass flow controller counterclockwise to establish flow. This will cause the gauge pressure to increase.

Electronic pressure control:

a. To change the units, press: gold 1 ENTER . Then press the number of the corresponding unit you want to use:

1 = psi
2 = bar
3 = kPa

b. The example below sets Inlet B (Injector B) pressure to 10 psi. Use the example to set the pressure you have selected.

c. Press:

gold INJ B PRES [illegible] [illegible] ENTER *Sets inlet B pressure to 10 psi*

	ACTUAL	SETPOINT
EPP B	10.0	10.0

The GC display looks like this

Note: To keep the pressure constant through an oven ramp program, see Chapter 10, *Using Electronic Pressure Control.*

2. Check the septum purge flow rate if you have an EPC system.

Excess carrier gas is vented through the septum purge vent. Although the septum purge vent is not adjustable, you should check the flow. Do not cap off the flow from the purge vent.

Carrier Gas Type	Approx. Flow
H_2	3.5-6.0 ml/min
He	1.5-3.5 mi/min
N_2	1.5-3.5 ml/min
Argon/Methane	1.5-3.5 ml/min

3. Set the linear velocity:

Use the timer feature (see *Using the internal stopwatch* in this chapter) and make repeated injections of an unretained component. Then adjust the column head pressure as necessary to obtain the expected retention time for the desired linear velocity,

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

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:

$$t_{r\ (expected)}\ (\text{in min}) = 1.67\ \frac{\text{Column Length (m)}}{\text{Linear Flow Velocity (cm/sec)}}$$

4. Use the following steps to set the splitless injection timetable:

Splitless injection is made possible by redirecting the inlet purge flow away from the inlet insert at the time of injection. After injection, sufficient time is allowed for solvent and sample components to reconcentrate at the head of the column. Then by redirecting the purge flow back through the insert, solvent vapor within the inlet insert is purged.

There are two purge control channels, one for inlet A and one for inlet B.

a. To redirect the purge flow away from the column to allow for a splitless injection:

Press: PURGE/VALVE A (or B) OFF .

Note: Flow through the insert at this point passes only through the column.

b. To restore the inlet purge flow:

Press: PURGE/VALVE A (or B) ON .

Manual purge switching:

Use the keyboard to switch the splitless solenoid valve on or off manually. Attempting to switch the valve on (or off) when it is already on (or off) has no effect.

The figure below shows typical displays for current valve status and for verifying the timed events table to switch the valve automatically during a run.

	ACTUAL	SETPOINT
INL PURGE A ON		

	ACTUAL	SETPOINT
INL PURGE B ON		

	ACTUAL	SETPOINT
PURGE B ON		1.50

	ACTUAL	SETPOINT
PURGE B OFF		0.10

Typical Split/Splitless Inlet Purge Displays

Automatic purge switching:

Use the steps below to switch the splitless solenoid valve on or off automatically once during a run. The valve remains in its final state after termination of the run.

a. To put the inlet into the splitless mode:

Press: PURGE/VALVE A (or B) OFF.

b. Set the on time value.

The on time value is the specific time delay after injection for the insert purging to occur. The on time value depends upon the components, solvent, injection volume, flow rate through the inlet, and internal insert volume (approximately 1 ml). Generally, an on time value between 0.6 and 1.5 minutes is reasonable.

Press: PURGE/VALVE A (or B) TIME ON
on time value ENTER.

c. Set the off time value.

The purge A (or B) off time value should be somewhat less than the total length of the time for the run. Inlet purge flow is switched off automatically just prior to the end of the run to ensure that the inlet valve is in the correct state for the start of the next run.

Press: PURGE/VALVE A (or B) TIME OFF
off time value ENTER.

d. To display the time during the run when the purging will be halted:

Press: PURGE/VALVE A (or B) TIME OFF

e. To display elapsed time during the run when purging will be restored:

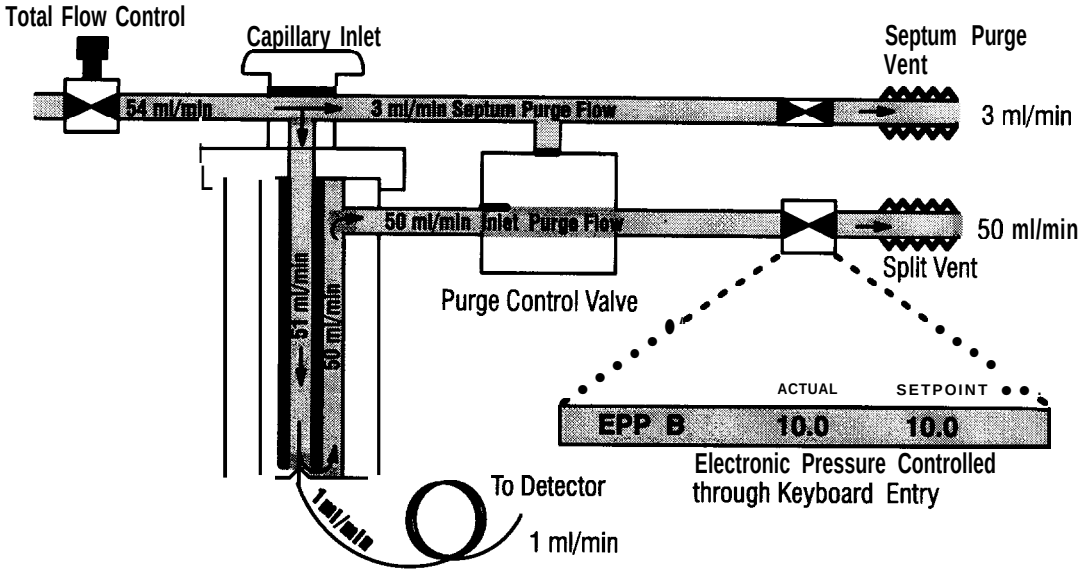
Press: PURGE/VALVE A (or B) TIME ON.

Once the inlet purging event is displayed, you can enter a new elapsed time (to 0.01 minute or similar) at any time. Elapsed time to halt inlet purging must be prior to injection (typically 0.00). Both on and off times are referenced to the start of the oven program.

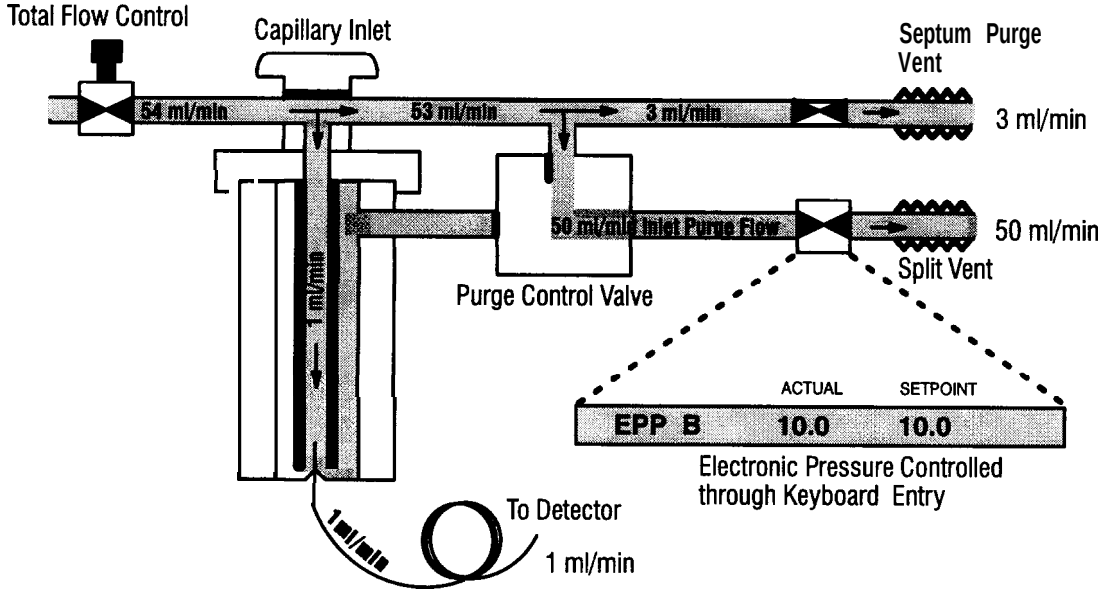
Note: Any entered value from 0.00 through 650.00 minutes is valid. However, the system ignores an entered time greater than that of the run time itself and does not switch the purge valve. The system also ignores the switch command if the programmed on and off times are the same.

For information about EPC (constant pressure, optimizing splitless injection), see Chapter 10, *Using Electronic Pressure Control.*

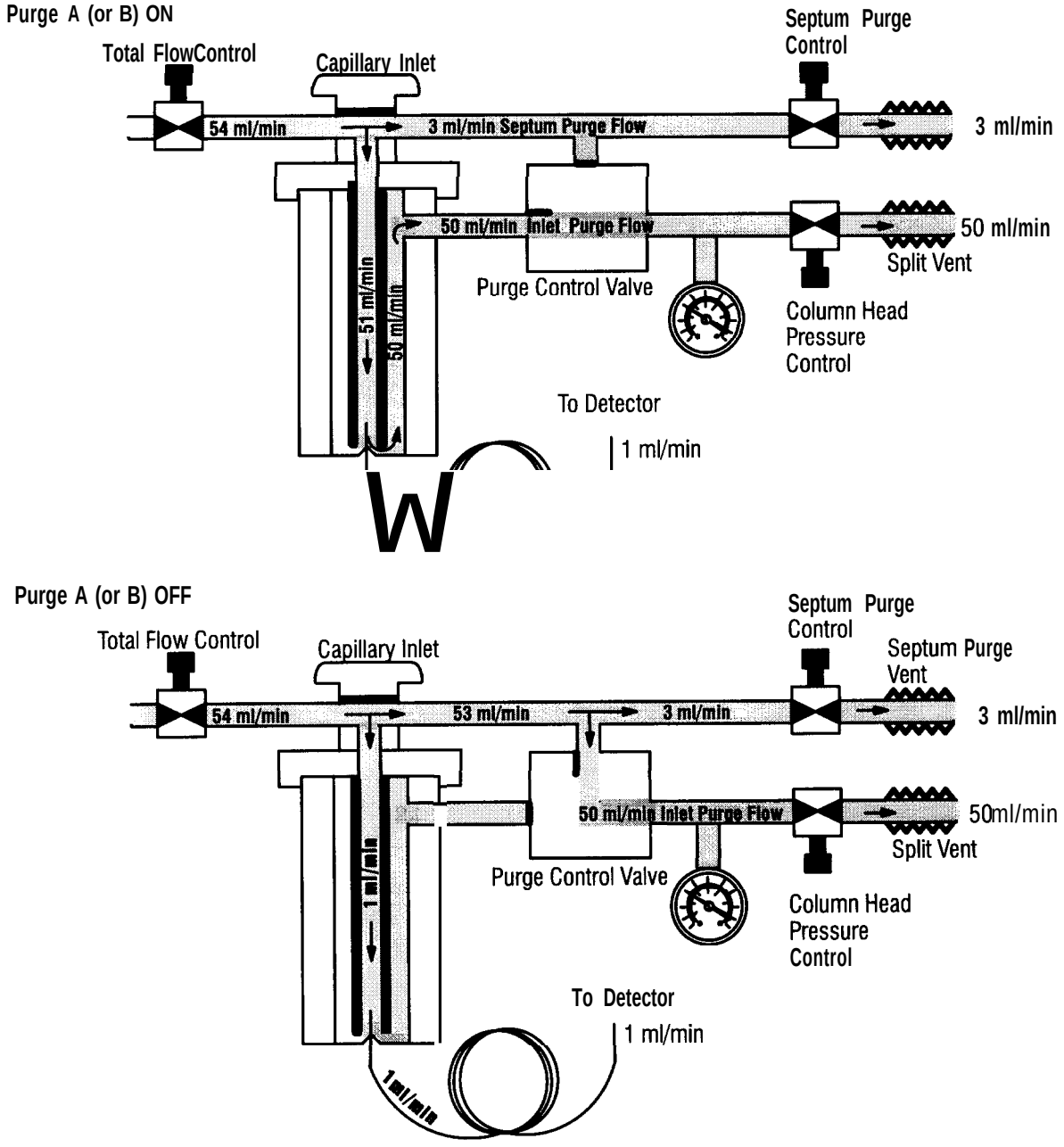
Purge A (or B) ON



Purge A (or B) OFF



Splitless Flow Diagram for Electronic Pressure Control



Splitless Flow Diagram for Manual Flow Control

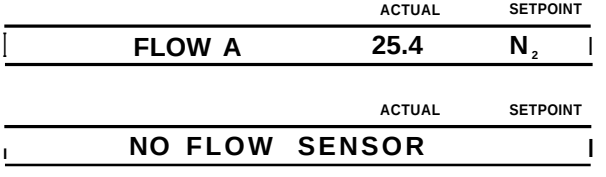
Displaying the gas flow rate

Note: If you have EPC, you will not have this feature.

If electronic flow sensing (EFS) is installed in the carrier gas system to the inlet, you can display total supply flow rate through the system. To display the total supply flow rate:

Press: FLOW A (or B).

(EFS cannot be used with EPC inlets.) Typical gas flow rate displays are shown below.



Typical Electronic Flow Rate Sensor Displays

Designating gas type

To scale the displayed flow rate value properly you must designate one of the four commonly used gases. Select the appropriate gas type from the table below.

Defining Type of Gas to Be Monitored

Number	Gas Type	Preferred Use
1	He (Helium)	TCD
2	N_2 (Nitrogen)	General
3	H_2 (Hydrogen)	Capillary
4	Ar/CH_4 (Methane in Argon)	ECD

Use the steps below to select one of these gases for a particular flow channel.

1. Press: FLOW A (or B) to display flow A (or B).
2. Press: 1, 2, 3, or 4 followed by ENTER.

The current flow rate is displayed and scaled appropriately for the chosen gas type.

To use a gas other than the four standard gases listed above, select the standard gas (He, N_2, H_2, or Ar-Me/CH_4) closest in thermal conductivity to the gas being used.

WARNING Do not pass any corrosive gas through the EFS.

The maximum usable range for H_2 is 100 ml/min. If flow rates above 100 ml/min are used, a gas other than He, N_2, or AR/CH_4 is being used, or maximum accuracy in displayed flow rate is required, you may need to calibrate the EFS. See the *HP 5890 Series II Reference Manual.*

Using the internal stopwatch

The stopwatch timer is useful for setting gas flow rates and measuring elapsed time between events of interest. In stopwatch mode, both time (to 0.1 second) and reciprocal time (to 0.01 min^{-1}) are displayed simultaneously. Use the following steps to access the stopwatch.

1. Press: TIME repeatedly until the stopwatch display appears.
2. Press: ENTER to start the stopwatch. Press: ENTER again to stop it.
3. Press: CLEAR to reset the stopwatch.

Stopwatch Mode

ACTUAL	SETPOINT
t=o:oo. o	1 /t = 0.00

Time Display

Operating Detector Systems

Operating Detector Systems

This chapter provides general and specific operating information for the five HP 5890 Series II and HP 5890 Series II Plus GC detector systems:

- Flame ionization detector (FID)
- Thermal conductivity detector (TCD)
- Nitrogen-phosphorus detector (NPD)
- Electron capture detector (ECD)
- Flame photometric detector (FPD)

Note: The Series 530 µ column supplied with the HP 5890 must be conditioned before use. This is done by establishing a flow of carrier gas at 30 to 60 ml/min through the column while the column is heated at 250°C for at least 4 hours. Refer to the *HP 5890 Series II Reference Manual, Preventive Maintenance.*

Displaying detector status

To turn a detector on or off, or to display the current status, press: DET A (or B). Pressing DET A (or B) while using a TCD toggles the polarity between positive and negative.

The following occurs when each detector is turned off:

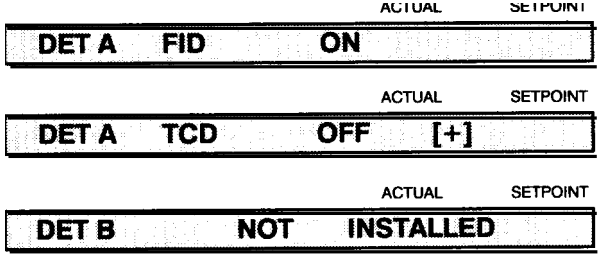
FID: The output signal and collector voltage are switched off. The flame, if already lit, remains so until gas supplies are turned off.

NPD: The output signal, its collector voltage, and the current through its active element are switched off.

ECD: The output signal is switched off.

TCD: The output signal, flow modulator valve, and filament current are switched off.

FPD: The output signal and high voltage are switched off. The flame remains lit until gas supplies are turned off.



Typical Detector Status Displays

Turning a detector on or off

Caution

The TCD filament can be permanently damaged if gas flow through the detector is off or interrupted while the detector is on. Make sure the detector is off whenever changes/adjustments are made affecting gas flows through the detector.

Once the desired detector is displayed, press [ON] to turn it on and [OFF] to turn it off. The change is immediately displayed.

Note that turning a detector off or on does not affect its zone temperature. Detector temperature is controlled separately through the temperature control key associated with the particular heated zone ([DET A TEMP] or [DET B TEMP]). For more information, see Chapter 3, *Setting Heated Zone Temperatures.*

Monitoring detector output

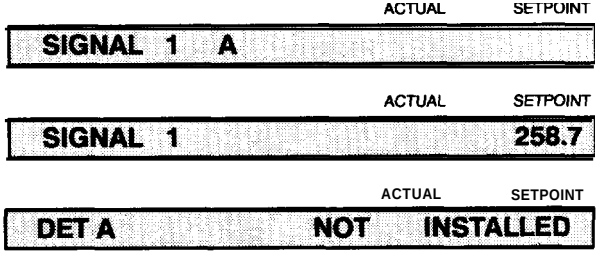
Knowing the detector output is particularly useful when you initialize the detector for operation, for example, when you light the flame for an FID, set the power to an NPD active element, or check noise (baseline frequency) for an ECD.

1. To display the detector output at any time, assign an output channel (signal 1 and/or
   signal 2 if installed) to a particular detector (identified by its location, A or B).

   Press: SIG 1 (or SIG 2) A (or B) ENTER .

2. Display the signal level for the detector by pressing SIG 1 (or SIG 2).
3. Press: SIG 1 (or SIG 2) again to display the signal source assigned to the particular output channel.

   The figure below shows typical displays:



Typical Displays for Monitoring the Detector Output Signal

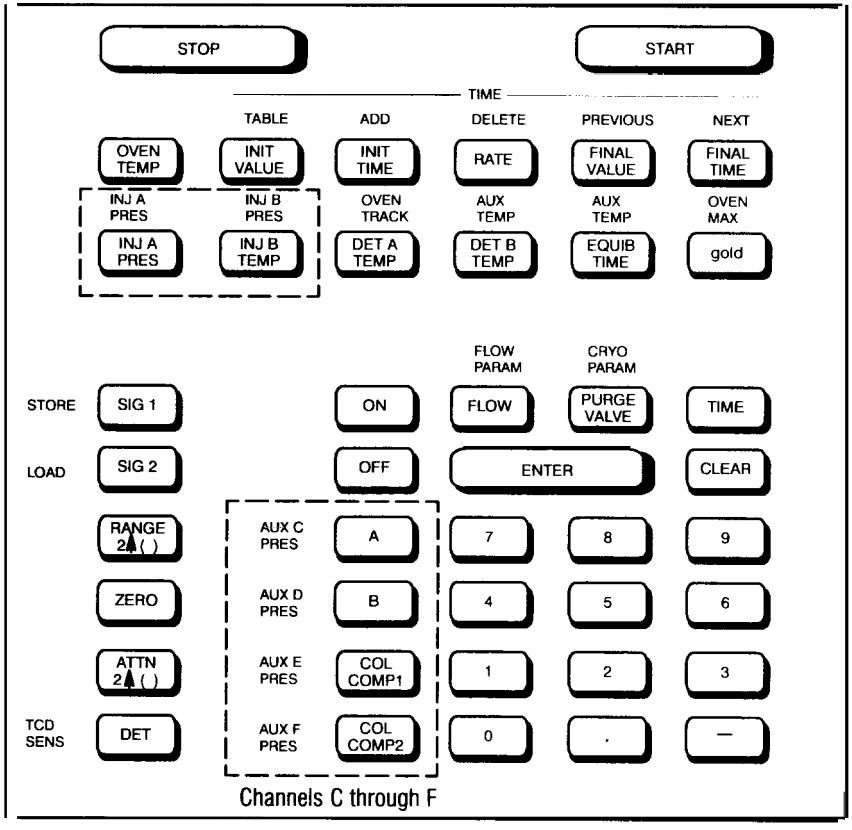
When detector A or B is not installed, signal 1 or 2 will be undefined. If you try to set a detector that is not installed, the display shows that it is not installed.

The display shows the signal in real time, reacting immediately to anything affecting detector response. This provides a convenient method for monitoring detector output.

Operating detectors using electronic pressure control

This section describes general operations for detectors with EPC. The next section describes how to set flows for systems controlled manually or electronically.

Electronic pressure control allows you to control all auxiliary gases that are configured using EPC from the keyboard of the HP 5890 GC. Electronic control is available on channels C through F for auxiliary gases. The figure below shows the HP 5890 GC keyboard, including the EPC keys that allow you to access channels C through F:



This section contains basic operating instructions for channels C through F, including:

- Accessing the auxiliary channels
- Zeroing the pressure channel for calibration
- Setting a constant pressure program
- Setting pressure ramps
- Changing pressure ramps
- Verifying pressure ramps

Accessing auxiliary channels C through F

The four EPC auxiliary channels are accessed through the existing GC keyboard by using the following keys:

Press:	To Access:
gold + A	*Auxiliary EPC channel C*
gold + B	*Auxiliary EPC channel D*
gold + COL COMP1	*Auxiliary EPC channel E*
gold + COL COMP2	*Auxiliary EPC channel F*

After accessing each channel, the GC display shows the channel you have selected and the actual and setpoint values. For example, after accessing auxiliary EPC channel C, the GC display might look like this:

	ACTUAL	SETPOINT
EPP C	**10.0**	**10.0**

Zeroing the pressure channel

When you zero the pressure, you are compensating for background pressure. The system is zeroed when it is shipped, but you should check it periodically, especially when the ambient laboratory temperature changes dramatically

Note: You should zero the EPC channels 30 to 60 minutes after the system has heated up, because changes in temperature may cause fluctuations while the instrument heats to its final temperature.

To zero the channel accurately all flow must be removed from the system before you enter the offset value. For each channel, you will first set the channel to zero, and then enter the value labeled actual as the offset. The following example zeroes channel C.

1. Turn off all inlet and detector gases.
2. Repressurize the inlet and detector gases to 0.0 psi.
3. Set the auxiliary EPC channel C pressure to zero:

   Press: gold B 0 . 0 ENTER.

	ACTUAL	SETPOINT
EPP C	5.0	0.0

The GC display looks like this.

4. Press: gold 4 ENTER 5 ENTER

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

	ACTUAL	SETPOINT
EPP C	5.0	5.0

The GC display looks like this.

   Follow the same procedure to zero the remaining auxiliary EPC channels. Remember that the system must be completely depressurized before entering the value labeled actual.

5. To zero channel D:

   a. Press: gold B 0 . 0 ENTER

   Set auxiliary channel D pressure to 0.0.

   b. Press: gold 5 ENTER *value* ENTER.

	ACTUAL	SETPOINT
EPP D	5.0	5.0

The GC display looks like this.

6. To zero channel E:

a. **Press:** gold COL COMP1 0 . 0 ENTER
Set auxiliary channel E pressure to 0.0.

b. **Press:** gold 6 ENTER *value* ENTER .

	ACTUAL	SETPOINT
EPP E	**5.0**	**5.0**

The GC display looks like this.

7. To zero channel F:

a. **Press:** gold COL COMP2 0 . 0 ENTER *Set auxiliary channel F pressure to 0.0.*

b. **Press:** gold 7 ENTER *value* ENTER .

	ACTUAL	SETPOINT
EFP F	5.0	5.0

The GC display looks like this.

Setting constant pressure

To set constant pressure for each of the four auxiliary channels, (where *value* is the desired constant pressure value):

Press: gold A *value* ENTER **for EPC channel C.**
Press: gold B *value* ENTER **for EPC channel D.**
Press: gold COL COMP1 *value* ENTER **for EPC channel E.**
Press: gold COL COMP2 *value* ENTER **for EPC channel F.**

Note: To enter a constant pressure for a run, the initial time in the program must be as long as (or longer than) the run time.

After setting the constant pressure for each channel, the GC display shows the channel you have selected and the actual and setpoint pressure values. For example, if you set EPC channel C to 60, the GC display looks like this:

	ACTUAL	SETPOINT
EPP C	*60.0*	*60.0*

Setting pressure ramps

To set pressure ramps for each of the four auxiliary channels (where value is the setpoint value for the specific part of the ramp):

To set the program for EPC channel C:

Press: gold A INIT VALUE *value* ENTER INIT TIME *value* ENTER RATE *value* ENTER FINAL VALUE *value* ENTER FINAL TIME *value* ENTER .

To set the program for EPC channel D:

Press: gold B INIT VALUE *value* ENTER INIT TIME *value* ENTER RATE *value* ENTER FINAL VALUE *value* ENTER FINAL TIME *value* ENTER .

To set the program for EPC channel E:

Press: gold COL COMP1 INIT VALUE *value* ENTER INIT TIME *value* ENTER RATE *value* ENTER FINAL VALUE *value* ENTER FINAL TIME *value* ENTER .

To set the program for EPC channel F:

Press: gold COL COMP2 INIT VALUE *value* ENTER INIT TIME *value* ENTER RATE *value* ENTER FINAL VALUE *value* ENTER FINAL TIME *value* ENTER .

Changing pressure ramps

To change pressure ramps for each of the four auxiliary EPC channels, follow the procedure for setting pressure ramps and enter new values for any of the variables.

Example of setting pressure ramps

This example shows how to enter a pressure ramp to program the gases for a detector that is installed in the A position and controlled by auxiliary channel D.

1. To access auxiliary channel D:

Press: gold B

	ACTUAL	SETPOINT
EPP D	10.0	10.0

The GC display looks like this.

2. Enter a pressure program for auxiliary channel D:

a. Press: INIT VALUE 40 ENTER to set an initial pressure of 40 psi.

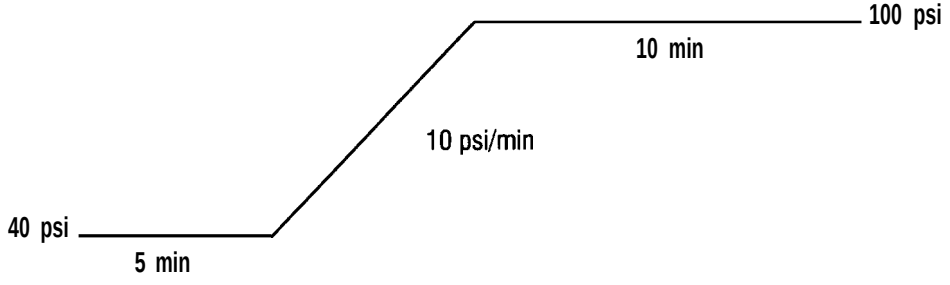
b. Press: INIT TIME 5 ENTER to maintain the initial pressure for 5 minutes.

c. Press: RATE 10 ENTER to increase the pressure by 10 psi per minute.

d. Press: FINAL VALUE 100 ENTER to set a final pressure of 100 psi.

e. Press: FINAL TIME 10 ENTER to maintain the final pressure for 10 minutes.

The system will now ramp the pressure as shown below:



Verifying pressure ramps

To verify pressure for each of the four auxiliary channels, access the pressure channel to display the actual and setpoint pressure values. For example:

Press: gold A to verify the pressure for auxiliary EPC channel C.

	ACTUAL	SETPOINT
EPP C	60.0	60.0

The GC display looks like this.

Setting capillary makeup gas flow rate

Capillary makeup gas is the gas that you add to the detector to compensate for the low carrier gas flow rates used for capillary columns. Low carrier gas flow must be compensated for because detectors are designed to operate best with a carrier flow rate of at least 20 ml/min, which is typical of packed-column GC applications. Carrier flow rates less than 10 ml/min (typically for capillary GC applications) require capillary makeup gas to ensure a total flow rate (carrier plus makeup) of at least 20 ml/min.

For the ECD, capillary makeup gas should be used even with HP Series 530 µ capillary columns because the detector requires high total flow rate (at least 25 ml/min).

Exceptions to makeup gas flow

The TCD requires a total flow rate of only 5 ml/min (with about 15 ml/min TCD reference flow). For the FID, TCD, NPD, and FPD, HP Series 530p capillary columns may be used without capillary makeup gas as long as the carrier flow rate is between 10 and 20 ml/min. Some loss of detector sensitivity may occur at lower flow rates.

For the FID, NPD, and FPD, makeup gas is added directly to hydrogen within the detector flow manifold. For an ECD or TCD, it is added into the column gas stream via a capillary makeup gas adapter fitted into the detector column inlet.

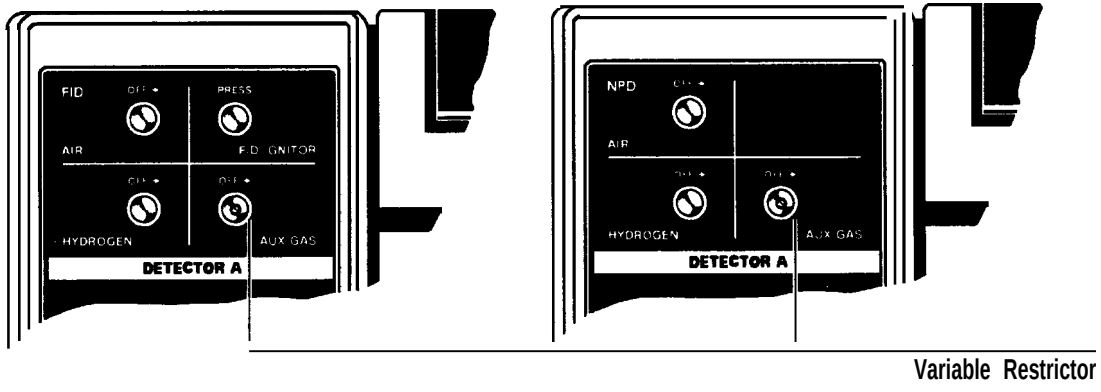
To set the makeup flow rate supply pressure for capillary makeup gas to about 276 kPa (40 psi):

1. Make sure the column and makeup gas fittings (if used) are properly installed.
2. Turn off all gas flows through the detector except the carrier flow.
3. Adjust the column flow to the desired value for the detector and column. Measure the flow at the detector exit with a bubble flow meter.
4. Use the following instructions to enter the makeup gas values for either manual or EPC systems:

Manual pressure control

a. **Set** supply pressure for capillary makeup gas to about 276 kPa (40 psi).

b. Open the auxiliary gas on/off valve. Use a small screwdriver to turn the variable restrictor at the center of the on/off valve as necessary to obtain the desired total flow rate (column plus makeup).



Variable Restrictor Adjustment

Electronic pressure control:

a. Set the supply pressure to the auxiliary EPC channel to 40 psi.

b. Open the Aux gas (makeup gas) on/off valve completely. You will use the auxiliary EPC pressure to control the auxiliary gas flow rate. Be sure to open the needle valve fully by turning it clockwise with a screwdriver.

c. Select the pressure units you would like to use.

To change the units, press: [gold] [1] [ENTER] . Then press the number of the corresponding unit you want to use:

[1] = psi
[2] = bar
[3] = kPa

d. The example below sets Inlet B (Injector B) pressure to 10 psi. Use the example to set the pressure you have selected.

e. Press:

[gold] [INJ B PRES] [1] [0] [ENTER] *Sets inlet B pressure to 10 psi.*

	ACTUAL	SETPOINT
EPP B	10.0	10.0

The GC display looks like this

Note: To keep the flow constant through an oven ramp program, see Chapter 10, *Using Electronic Pressure Control.*

f. Adjust the makeup gas pressure to the detector as necessary to *obtain 30* ml/min total flow rate (column plus makeup).

5. Refer to the appropriate detector section to initialize the detector for operation.

If the power fails . . .

If the power fails frequently, turn off the detector whenever it is not in use.

Note: When a detector is turned on after being off, it must be given time to stabilize before it can be used at high sensitivity. The baseline will drift until the detector reaches equilibrium.

When power is restored after a power failure, the detector recovers to the same state as when the power failed. The active element is restored to "on" if it was on before the power failure.

If the gases used to light the FID or FPD are controlled with EPC, the flow will go to zero when the power fails and return to setpoint when restored. You will need to relight the flame after the power is turned on. For auxiliary EPC, the GC returns to the setpoints it had before the power failed.

Shutting down each day

On a daily basis, use the steps in the following procedure to shut down the detector:

1. In most cases, leave the detector on and at operating temperature to avoid a long equilibration time at startup.
2. Leave the carrier flow onto protect the column(s). For extended shutdown periods, cool the oven to room temperature, and then turn the carrier flow off.
3. With EPC applications, you can reduce the gas flows to conserve gas and still have the detector lit and ready.

   Note: For more information about the Gas Saver application, see Chapter 10, *Using the Gas Saver Application*.
4. With the ECD, you may want to reduce the sensitivity by lowering the temperature to prolong its lifetime. For extended shutdown periods, cap off the column interface and leave a small amount of makeup gas flowing through the system.

Operating the flame ionization detector (FID)

The flame ionization detector (FID) responds to compounds that produce ions when burned in an H_2-air flame. These include all organic compounds, although a few (such as formic acid and formaldehyde) exhibit poor sensitivity. This selectivity can be advantageous-for example, when used as solvents, H_2O and CS_2 do not produce large solvent peaks.

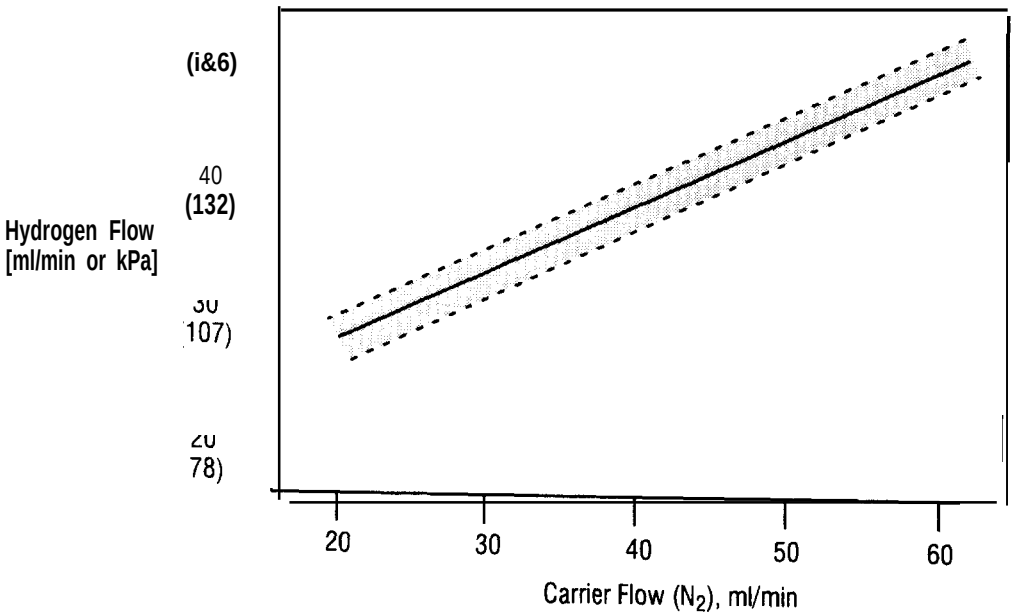
Compounds Producing Little or No Response

Permanent gases	CO
Nitrogen oxides	CO_2
Silicon halides	CS_2
H_2O	O_2
NH_3	CCl_4*

*Measured at the jet tip

The system is linear for most organic compounds from the minimum detectable limit through concentrations greater than 10^7 times the minimum detectable limit. Linear range depends on each specific compound and is directly proportional to sensitivity of the FID toward the given compound.

For maximum sensitivity, optimize the flows using standard samples containing components of interest in expected concentrations. Use the standard to experiment with different carrier, air, and H_2 flow rates, and determine the flow rates giving maximum response.



Optimum Response Hydrogen versus Carrier Flow

In general, where sample components of interest are in high concentration, increased air flow may be necessary (up to 650 ml/min). Where components of interest are in low concentration, reduced air flow rates are acceptable (375 to 425 ml/min).

Setting up the FID for operation

To setup the FID for operation, you must do the following:

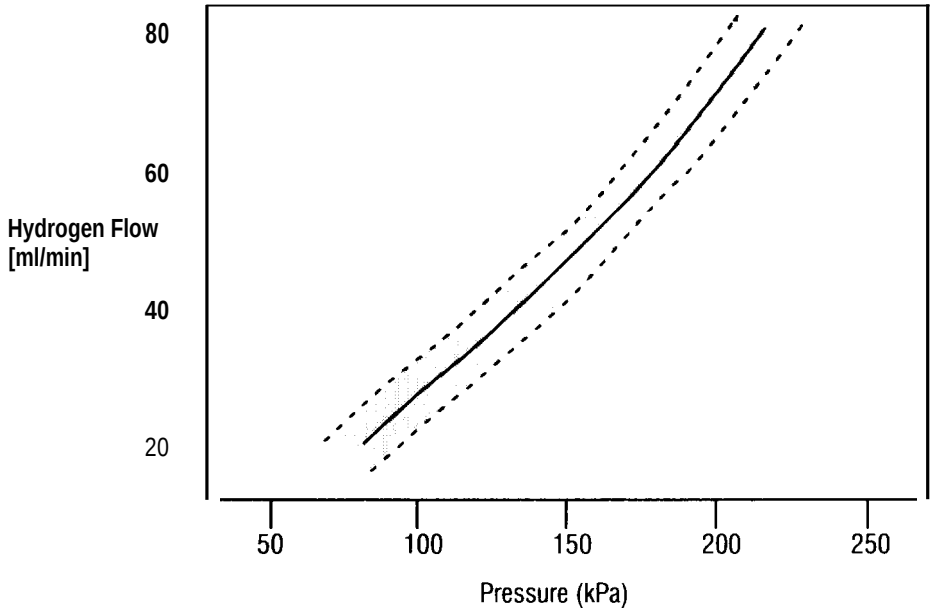
. Set the flow (for either packed or capillary columns).

. Set the detector flow rates.

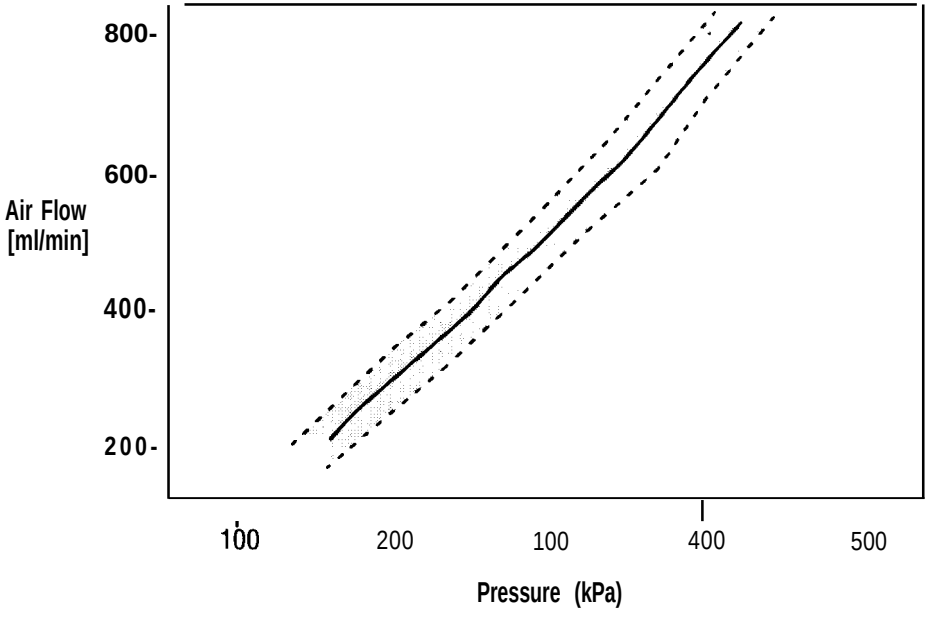
. Turn on the detector.

. Ignite the flame.

Hydrogen Flow Rate versus Pressure



Air Flow Rate versus Pressure



Setting the FID flow for packed columns

The gas flow rates given in this section ensure good, reliable detector behavior for most applications. To optimize detector behavior for a specific application, use a standard sample matched to the application and experimentally try other flow rates.

WARNING Flame ionization detectors use H_2 gas as fuel. If H_2 flow is on and no column is connected to the detector inlet fitting, H_2 gas can flow into the oven and create an explosion hazard. Inlet fittings must have either a column or a cap connected at all times that H_2 is supplied to the instrument.

Note: Depending upon the column type used and the analyses to be performed, you may have to change the jet in the FID.

Use the steps in the following procedure to set the FID flow in a packed column. This procedure assumes that detector support gases are connected, the system is leak-free, the correct jet is installed, and a column is installed.

1. Close the Aux Gas on/off valve. This controls the makeup gas.
2. Set the column flow rate to 30 ml/min. Because the procedure for setting column flow rate depends on the column installed and the inlet system used, refer to the appropriate inlet system information in Chapter 4.
3. **Set** the oven and heated zones to the desired operating temperatures.
4. Gently close the on/off controls for H_2 and air by turning them clockwise.
5. Using the flow rate versus pressure figures (shown in this section) and the carrier gas flow rate, set supply pressures for H_2 and air to obtain the correct flow rates (30 ml/min of H_2 and 430 ml/min of air are correct for most applications).

You can set H_2 and air flow rates simply by setting their respective pressures. However, if flow rates need to be verified, continue with this section using the bubble flow meter. Otherwise, open the H_2 and air on/off valves by turning them fully counterclockwise, and proceed to *Igniting the Flame* later in this section.

6. Use the following steps to set the H_2 flow rate to 30 ml/min:

   a. Attach a bubble flow meter to the FID collector.

WARNING To minimize risk of explosion when using a bubble flow meter, never measure air and H_2 together. measure them separately.

   b. Open the H_2 on/off valve by turning it counterclockwise. Measure the total flow rate (column plus H_2) through the detector.

   c. Adjust the H_2 pressure to the detector to obtain a total flow rate (column plus H_2) of about 30 ml/min.

   d. Close the H_2 on/off valve.

7. Use the following steps to set the air flow rate to 400 ml/min:

   a. Open the air on/off valve by turning it counterclockwise. Measure the total flow rate (column plus air) through the detector.

   b. Adjust the air pressure to the detector to obtain a total flow rate (column plus air) of about 430 ml/min.

8. Remove the bubble flow meter from the FID collector.

9. Open the H_2 on/off valve. Proceed to *Igniting the Flame* later in this chapter.

Setting the FID flow for capillary columns

WARNING Flame ionization detectors use H_2 gas as fuel. If H_2 flow is on and no column is connected to the detector inlet fitting, H_2 gas can flow into the oven and create an explosion hazard. Inlet fittings must have either a column or a cap connected at all times that H_2 is supplied to the instrument.

Note: Depending upon the column type used and the analyses to be performed, you may have to change the jet in the FID.

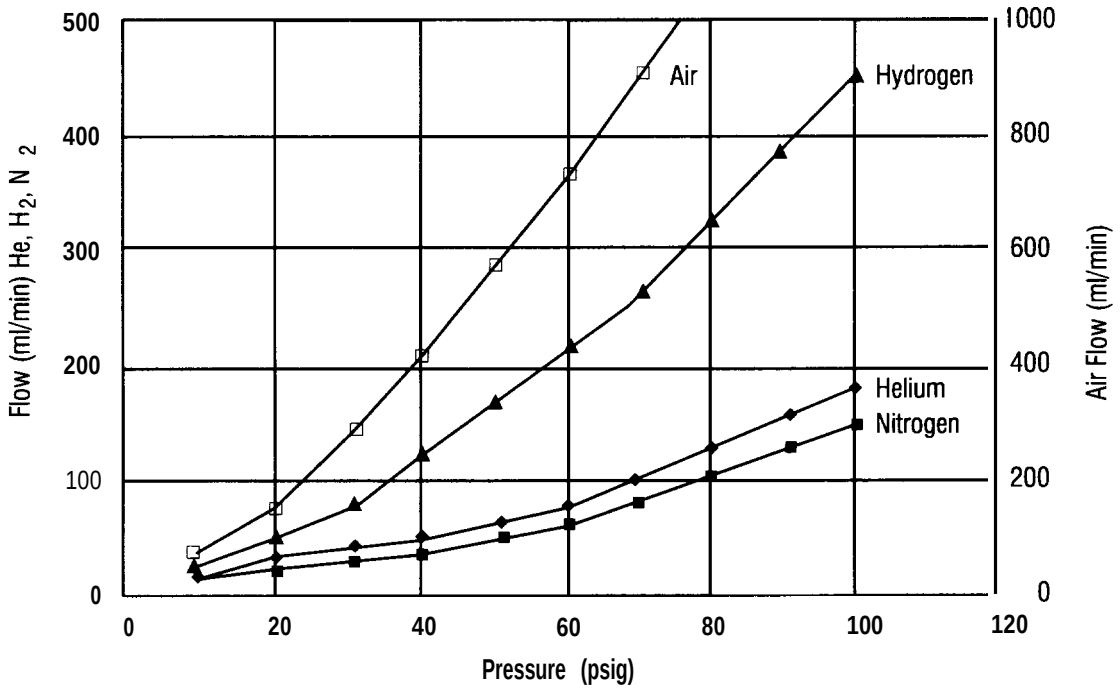
The following table and graph show the optimal flow rates at which to control your FID.

Typical Pressure versus Flow for FID Flow Restrictors
Values computed using ambient temperature of21‘C and pressure of 14.56 psi

Flow Restrictor Pressure		FID Makeup HP pn 19243-60540 Green and **Red Dots** Flow (ml/min)		FID Hydrogen HP pn 19231-60770 Red Dot Flow (ml/min)	FID Air HP pn 19231-60610 Brown Dot Flow (ml/min)
kPa	psig	Nitrogen	Helium	Hydrogen	Air
69.0	**10.0**	6.4	7.2	18.0	65.0
137.9	20.0	15.0	17.0	44.0	157.0
206.8	30.0	26.0	29.0	77.0	273.0
275.8	40.0	39.0	44.0	116.0	410.0
344.7	50.0	53.0	61.0	162.0	561.0
413.7	60.0	69.0	80.0	211.0	726.0
482.6	70.0	86.0	100.0	264.0	900.0
551.6	80.0	104.0	122.0	322.0	1084.0
620.5	90.0	123.0	150.0	383.0	
689.5	100.0	143.0	178.0	445.0	

= Recommended calibration points for using **EPC with the HP 3365 ChemStation**

FID Restrictors



Use the steps in the following procedure to set the column flow in a capillary column. This procedure assumes that the detector support gases are connected, the system is leak-free, the correct jet is installed, and a column is installed.

1. **Set** the column flow to the desired rate. Because the procedure for setting column flow rate depends on the column installed and the inlet system used, refer to the appropriate inlet system information in Chapter 4.
2. Set the oven and heated zones to the desired operating temperatures.
3. Adjust the carrier and makeup gas flow rate (column plus makeup) through the detector to at least 30 ml/min.

4. Use the following steps to set manually or verify the H_2 flow rate to approximately 30 ml/min:

WARNING

To minimize risk of explosion when using a bubble flow meter, never measure air and H_2 together. Measure them separately.

a. Attach the bubble flow meter to the FID collector.

b. Open the H_2 on/off valve by turning it counterclockwise. Measure the total flow rate (column plus makeup plus H_2) through the detector.

c. Adjust the H_2 pressure to the detector to obtain a total flow rate (column plus makeup plus H_2) of about 60 ml/min.

d. Close the H_2 on/off valve.

5. Use the following steps to set the air flow rate to 400 ml/min:

a. Open the air on/off valve by turning it counterclockwise. Measure total flow rate (column plus makeup plus air) through the detector.

b. Adjust the air pressure to the detector to obtain a total flow rate (column plus makeup plus air) of about 400 ml/min.

6. Remove the bubble flow meter from the FID collector.

7. Open the H_2 on/off valve and ignite the flame.

8. Use the following instructions to enter the makeup gas values for either manual or electronic systems.

Manual pressure control:

a. Set the supply pressure for the capillary makeup gas to about 276 kPa (40 psi).

b. Open the Aux gas (makeup gas) on/off valve by turning it counterclockwise.

c. Use a small screwdriver to turn the variable restrictor at the center of the on/off valve as necessary to obtain 30 ml/min total flow rate (column plus makeup).

Electronic pressure control:

a. Set the supply pressure to the auxiliary EPC channel to 40 psi using the keyboard.

b. Open the Aux gas (makeup gas) on/off valve. Turn the variable restrictor fully counterclockwise. Then adjust the pressure to set the desired flow rate.

c. Select the pressure units you would like to use.

To change the units, press: gold 1 ENTER . Then press the number of the corresponding unit you want to use:

1 = psi
2 = bar
3 = kPa

d. With auxiliary EPC, makeup gas can be controlled through auxiliary pressure channels C, D, E, or F from the keyboard. The example below sets the auxiliary channel C pressure to 10 psi.

Press:

gold A 1 0 ENTER *Sets auxiliary channel C pressure to 10 psi.*

	ACTUAL	SETPOINT
EPP C	**10.0**	**10.0**

The GC display looks like this.

Note: To keep the pressure constant through an oven ramp program, see Chapter 10, *Using Electronic Pressure Control.*

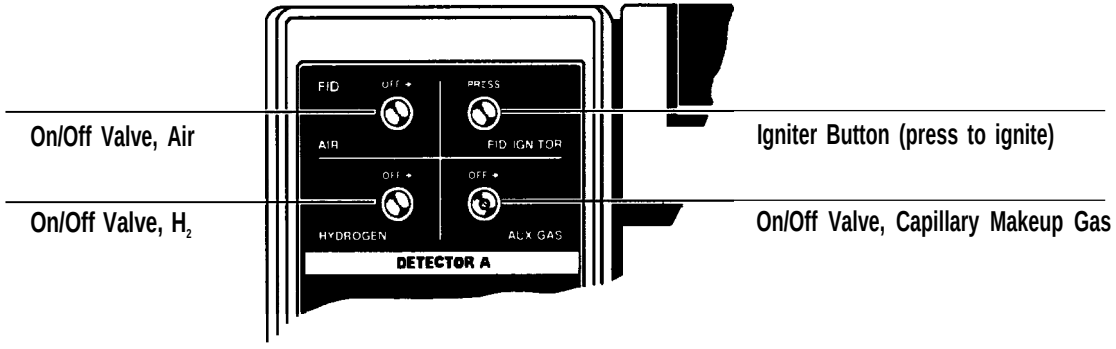
e. Adjust the makeup gas pressure to the detector as necessary to obtain 30 ml/min total flow rate (column plus makeup).

9. Gently close the on/off controls for H_2 and air by turning them clockwise.

10. Use the flow rate versus pressure graphs shown earlier in this chapter and the carrier gas flow rate to set the supply pressures for H_2 and air. Set the supply pressures to obtain the correct flow rates. (30 ml/min of H_2 and 400 ml/min of air are correct for most applications.)

Generally you can set H_2 and air flow rates simply by setting their respective pressures. For an explanation of the relationship of flow to

pressure in an EPC system, see Chapter 10, *Using Electronic Pressure Control.*



Flow Panel for Controlling FID Operation

Setting the makeup gas flow rate

Detectors are designed to operate best with a carrier flow rate of at least 20 ml/min, which is typical of packed-column GC applications. Carrier flow rates of less than 10 ml/min (typically capillary GC applications) require capillary makeup gas to ensure a total flow rate (carrier plus makeup) of at least 20 ml/min.

FID sensitivity depends on the ratio of H_2 to carrier flow (or carrier plus makeup gas for capillary columns). Use the procedure described in the following section to obtain maximum sensitivity.

You can set makeup flow rate manually or electronically. In both cases, good laboratory practice suggests that you calibrate the system with a bubble flow meter.

To set the makeup flow rate, set the supply pressure for capillary makeup gas to about 276 0kPa (40 psi):

1. Make sure the column and makeup gas fittings (if used) are properly installed.
2. Turn off all gas flows through the detector except the carrier flow.

3. Adjust the column flow to the desired value for the detector and column. Measure the flow at the detector exit with a bubble flow meter.

4. Use the following instructions to enter the makeup gas values for either manual or electronic systems.

Manual pressure control:

a. Set the supply pressure for the makeup gas to about 276 kPa (40 psi).

b. Open the Aux gas (makeup gas) on/off valve by turning it counterclockwise.

c. Use a small screwdriver to turn the variable restrictor at the center of the on/off valve as necessary to obtain the desired total flow rate (column plus makeup).

Electronic pressure control:

a. Set the supply pressure to the auxiliary EPC channel to 40 psi using the keyboard.

b. Open the Aux gas (makeup gas) on/off valve. Turn the variable restrictor fully counterclockwise. Then adjust the pressure through the keyboard to set the desired flow rate.

c. Select the pressure units you would like to use.

To change the units, press: gold 1 ENTER . Then press the number of the corresponding unit you want to use:

1 = psi
2 = bar
3 = kPa

d. The example below sets the auxiliary channel C pressure to 10 psi.

Press:

gold A 1 0 ENTER *Sets auxiliary channel C pressure to 10 psi*

	ACTUAL	SETPOINT
EPP C	10.0	10.0

The GC display looks like this

Note: To keep the flow constant through an oven ramp program, see Chapter 10, *Using Electronic Pressure Control.*

e. Adjust the makeup gas pressure to the detector as necessary to obtain 30 ml/min total flow rate (column plus makeup).

Turning the FID on and off

After the FID flows have been set, you can turn on the detector electronics.

To turn the FID on, press: DET A (or B) ON

To turn the FID off, press: DET A (or B) OFF

Igniting the FID flame

This procedure assumes that detector support gases are connected, the system is leak-free, the correct jet is installed, a column is installed, and the carrier gas and detector support gases have been set and verified at the detector exhaust vent.

1. Open the air, H_2, and makeup gas on/off valves.

Note: When using He as the capillary makeup gas, it maybe necessary to turn off the makeup gas flow temporarily until the flame is lit.

2. Before pressing the igniter button, enter the following:

DET A (or B) ON

SIG 1 (or SIG 2) A (or B) ENTER

SIG 1 (or SIG 2)

3. Press the igniter button.

Note: You can light the FID flame regardless of whether the detector is electronically on or off.

The displayed FID signal level will be in the range from 0 to 0.3 pA. When the flame lights, the displayed signal increases to some greater steady value (for example, 10 pA), indicating that the detector is active. The precise value depends upon the column and operating conditions. Turn makeup gas on if necessary.

You may also test for ignition by holding a cold, shiny surface (such as a chrome-plated wrench) over the collector exit. Steady condensation indicates that the flame is lit.

Operating the thermal conductivity detector (TCD)

This section assumes that all detector support gases are connected, leak-free, and that a column is installed.

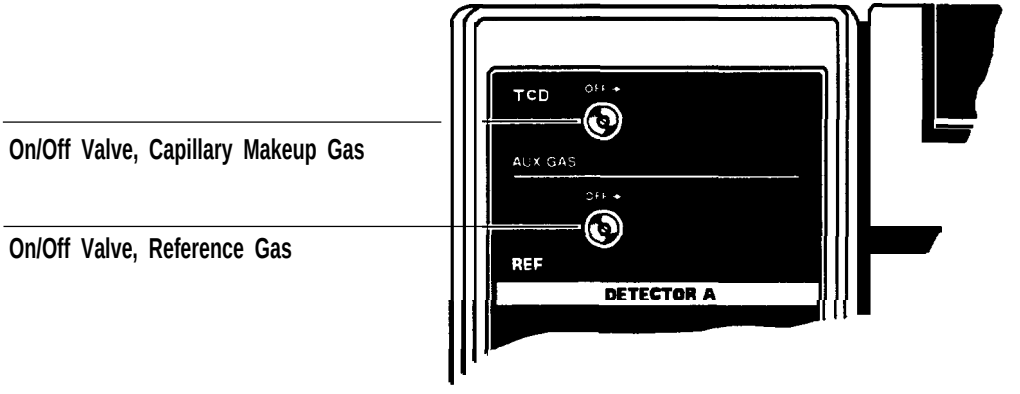
Caution

The TCD filament can be permanently damaged if gas flow through the detector is interrupted while the filament is operating. Make sure the detector is off whenever changes and adjustments are made affecting gas flows through the detector.

Also, exposure to 0_2 can permanently damage the filament. Make sure the entire flow system associated with the TCD is leak-free and that carrier/reference gas sources are uncontaminated before turning on the detector. Do not use Teflon tubing, either as column material or as gas supply lines, because it is permeable to 0_2.

When measuring TCD flow rates, attach a bubble flow meter directly to the detector exhaust vent using a small piece of rubber tubing as an adapter.

Note: When measuring TCD flow rates, a bubble flow meter is attached directly to the detector exhaust vent using a small piece of rubber tubing as an adapter. For convenience, the HP 5890 provides a stopwatch feature (see Chapter 4, *Using the Internal Stopwatch).*



Flow Panel Controlling TCD Operation

Setting up the TCD for operation

To setup the TCD for operation, you must do the following:

- Set the flow (for either packed or capillary columns).
- Set the carrier gas type.
- Set the sensitivity.
- Turn on the TCD.

This section will also show you how to:

- Invert TCD polarity.
- Use single-column compensation (SCC).

Setting the TCD flow for packed columns

The gas flow rates given in this section ensure good, reliable detector behavior for most applications. To optimize detector behavior for a specific application, use a standard sample matched to the application and experiment with other flow rates.

Use the steps in the following procedure to set the TCD flow in a packed column. This procedure assumes that detector support gases are connected, the system is leak-free, and a column is installed.

1. Set the detector zone temperature to the desired value (30 to 50°C greater than the maximum oven temperature to prevent sample condensation).

   Press: DET A TEMP (or DET B TEMP) *temp value* ENTER

2. Set the column flow rate to 30 ml/min. Because the procedure for setting column flow rate depends on the column installed and the inlet system used, refer to the appropriate inlet system information in Chapter 4.

   Note: When measuring column flow rate, make sure the reference gas flow through the detector is turned off (clockwise).

3. Use the following steps to set the reference gas flow rate.

   Note: A good guideline is to set the reference flow rate at 1.5 times the column flow rate.

   a. Open the on/off valve for the TCD reference gas flow by turning it counterclockwise.

   b. Use a small screwdriver to turn the variable restrictor at the center of the TCD reference gas on/off valve as necessary to obtain the desired flow rate (45 ml/minis correct when total flow is 30 ml/min).

4. If not already done, set the carrier gas type and detector sensitivity as discussed later in this chapter.

Setting the TCD flow for capillary columns

Use the steps in the following procedure to set the TCD flow in a capillary column. The gas flow rates given in this section ensure good, reliable detector behavior for most applications. To optimize detector behavior for a specific application, use a standard sample matched to the application and experiment with other flow rates.

1. Set the detector temperature to the desired value (30 to 50 'C greater than the maximum oven temperature to prevent sample condensation).

   Press: DET A TEMP (or DET B TEMP) *temp value* ENTER

2. Set the column flow to 1 to 2 ml/min. Because the procedure for setting column flow rate depends on the column installed and the inlet system used, refer to the appropriate inlet system information in Chapter 4.

   Note: When measuring column flow rate, make sure the reference gas flow through the detector is turned off (clockwise).

3. Set the makeup gas so that the total flow rate (column plus makeup) through the detector is 5 ml/min. Turn off the reference gas while making this measurement.

   When you use makeup gas, you should push the column all the way up into the detector and then pull it out approximately 1 mm. However, when you use a relatively high flow rate (and no makeup gas), the column should be only 1 to 2 mm above the ferrule. If you want to position the column all the way up for maximum inertness, then continue to use makeup gas and set it to 1 to 2 ml/min.

   Because a portion of the column passes through the TCD heated block and into the cell itself do not set the zone temperature for the TCD greater than the maximum temperature allowed for the column. A higher zone temperature may cause column bleed.

4. Use the following instructions to enter the makeup gas values for either manual or electronic systems. You can also control the TCD reference gas using the same steps used to control the makeup gas.

   Manual pressure control:

   a. **Set** the supply pressure for capillary makeup gas to about 276 kPa (40 psi).

   b. Open the Aux gas (makeup gas) on/off valve for TCD makeup gas flow by turning it counterclockwise.

   c. Use a small screwdriver to turn the variable restrictor at the center of the TCD makeup gas as necessary to obtain 5 ml/min.

      After the makeup gas is adjusted, the reference gas should be at least three times the total flow rate from the column plus makeup. Therefore, if the column plus the makeup flow is 5 ml/min, the reference flow equals 15 ml/min.

d. Open the on/off valve for the TCD reference gas flow by turning it counterclockwise.

e. Use a small screwdriver to turn the variable restrictor at the center of the TCD reference gas on/off valve as necessary to obtain 15 ml/min.

Electronic pressure control:

a. Set the supply pressure to the auxiliary EPC channel to 40 psi using the keyboard.

b. Open the Aux gas (makeup gas) on/off valve. Turn the variable restrictor fully counterclockwise. Then set the pressure through the keyboard to get the desired flow rate.

c. Select the pressure units you would like to use.

To change the units, press: gold 1 ENTER . Then press the number of the corresponding unit you want to use:

1 = psi
2 = bar
3 = kPa

d. The example below sets the auxiliary channel C pressure to 10 psi.

Press:

gold A 1 0 ENTER *Sets auxiliary channel C pressure to 10 psi*

	ACTUAL	SETPOINT
EFP C	10.0	10.0

The GC display looks like this

e. Adjust the makeup gas pressure to the detector as necessary to obtain 30 ml/min total flow rate (column plus makeup).

5. Set the reference gas flow rate:

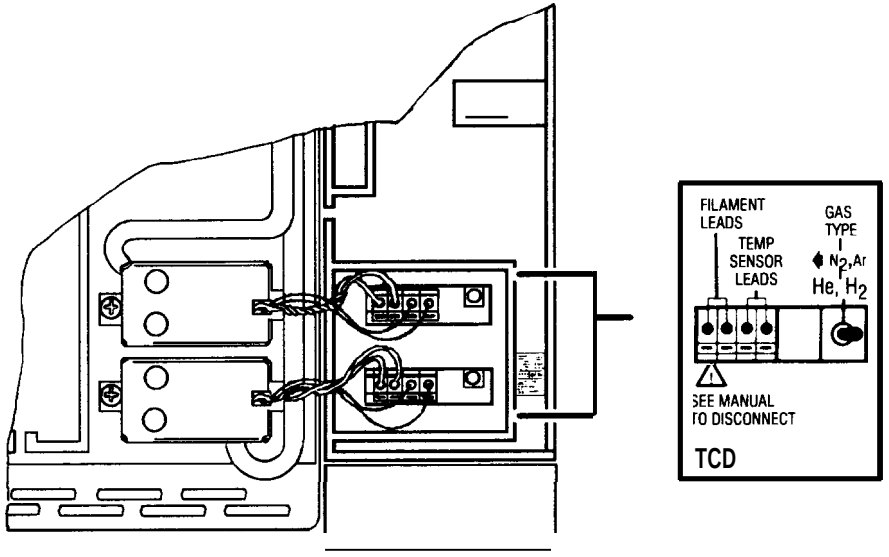
a. Open the on/off valve for the TCD reference gas flow by turning it counterclockwise.

b. Use a small screwdriver to turn the variable restrictor at the center of the TCD reference gas on/off valve as necessary to obtain the required flow.

6. If it is not already done, set the carrier gas and detector sensitivity as discussed in the following sections.

Setting the TCD carrier gas type

To optimize the detector sensitivity with respect to the carrier gas used, a switch is provided on the TCD signal board that is accessed at the top of the instrument under the top right cover.



1. Locate the switch and place it in the position appropriate for the carrier gas used (either N_2, Ar, or He, H_2).
2. To ensure the full dynamic range for the TCD, the reference gas (and capillary makeup gas, if used) must be the same as the carrier gas. Using different gases results in baseline offset.

Caution

The TCD filament can be permanently damaged if gas flow through the detector is interrupted while the detector is on. Make sure that the detector is off whenever changes and adjustments are made affecting gas flows through the detector.

Setting the TCD sensitivity

Two sensitivity (signal amplification) settings are available through the keyboard. The high- sensitivity setting increases sensitivity (area counts observed) by a factor of 32 and is usable in applications where component concentrations are less than 10 percent. Components that are more concentrated may exceed the output range for the TCD, causing flat-topped peaks. If this occurs, use the low-sensitivity setting instead.

You can change the sensitivity setting at any time without turning the detector off. Changing the setting has no effect upon filament lifetime. To set the TCD sensitivity from low to high:

Press: [gold] [DET] [A] (or [B]) [OFF] to set the TCD sensitivity to low.

Press: [gold] [DET] [A] (or [B]) [ON] to set the TCD sensitivity to high.

For information on how to change TCD sensitivity during a run, see Chapter 7, *Making a Run.*

Turning the TCD on and off

Caution

The TCD filament can be permanently damaged if gas flow through the detector is interrupted while the detector is on. Make sure the detector is off whenever changes/adjustments are made affecting gas flows through the detector.

After TCD flows have been set, the detector maybe turned on.

To turn the TCD on, press: [DET] [A] (or [B]) [ON].

To turn the TCD off, press: [DET] [A] (or [B]) [OFF].

Allow about l/2-hour for thermal stabilization (after the oven and zones achieve desired setpoint values) before using the TCD.

Inverting the TCD polarity

For information on inverting the TCD signal polarity refer to Chapter 6, *Controlling Signal Output.*

Using single-column compensation (SCC)

Because the TCD operates with only a single column, which is the analytical column, single-column compensation (SCC) is strongly recommended to achieve optimum baseline stability, particularly in temperature-programmed operation.

Alternatively, if two TCDs are installed, conventional dual-column compensation may be performed by defining the output signal as A-B or B-A so as to output a different signal from the two detectors. This assumes that the two detectors are operated using identical columns, temperatures, and flow rate conditions.

The HP 5890 allows you to perform a chromatographic blank run (run made with no sample injected) and stores the data as a baseline profile. The baseline profile must be consistent from run to run so it can be subtracted from the sample run data to remove baseline drift (usually caused by column bleed).

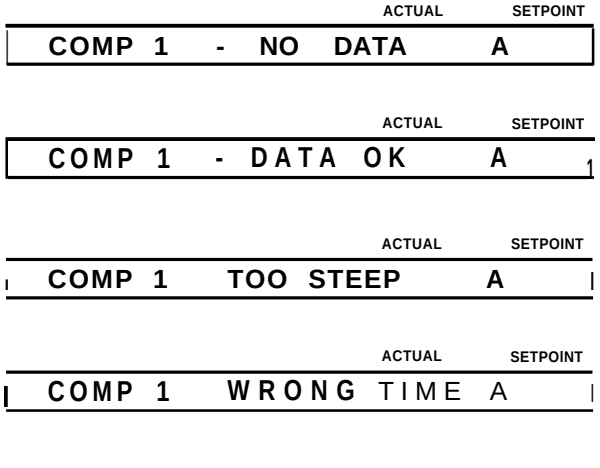
Note: Single-column compensation data is valid only for a specific detector and column combination, operating under defined temperature and gas flow rate conditions. Invalid results will occur if conditions by which blank run data is collected are different from conditions used to collect sample run data.

Two separate profiles may be stored as designated by COL COMP1 and COL COMP2. For example, you may store one each for two different detectors or two profiles for the same detector (using different chromatographic conditions).

Note: The STOP key is always active during a column compensation run if you need to abort the run at the HP 5890 GC.

Displaying the column compensation status

The status of column compensation data is displayed by pressing either COL COMP1 or COL COMP2. The figure below shows examples:



No baseline profile data is presently stored for detector **A** in **COMP 1.**

Valid baseline profile data is presently stored for detector **A** in **COMP 1.**

Change in baseline slope exceeds maximum value permitted. Column compensation data may not be valid.

Column compensation run aborted prematurely via STOP. Column compensation data may not be valid.

(Equivalent displays are possible for COMP 2 and/or detector B)

Typical Column Compensation Status Displays

In each display, COMP 1 or COMP 2 echoes the key pressed (COL COMP1 or COL COMP2). A or B indicates the assigned detector.

Initiating a column compensation run

After entering the oven temperature program to be used for later sample runs, a column compensation run is initiated by first pressing either [COL COMP1] or [COL COMP2] to display current column compensation status and to designate where the new baseline profile is to be stored.

. If the desired detector (A or B) is displayed, the column compensation run is initiated simply by pressing [ENTER].

. If the wrong detector is displayed, press either A or B to assign the desired detector; then press [ENTER] to initiate the column compensation run.

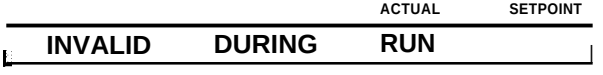
. [ ] followed by [ENTER] initiates two parallel column compensation runs, using the same oven temperature program and storing a baseline profile for each of the assigned detectors simultaneously.

This option is useful for sample analyses made using different detectors and/or columns but using identical temperature programs.

Note: A device connected via the remote start/HP 5890 ready cable that is started from the HP 5890 by a normal analytical run is not started by a column compensation run.

Additional details concerning functions available at the remote receptacle are found in the *HP 5890 Series II Site Prep and Installation Manual.*

Messages listed in the next figure are displayed either while a column compensation run is in progress or if there is a problem preventing the compensation run from starting.

	**Comp** run in progress. In this example, data from detector A is stored as **COMP 1** (accessed via COL COMP1).
	Displayed if an attempt to start a column compensation run is made while a sample run is in progress. No column compensation run is performed.
	The oven is not on. Once the oven is switched on, the column compensation run begins automatically when the oven is equilibrated at its initial temperature setpoint.
	An oven tempreurprogramis not defined: nonzero RATE setpoint value(s) must be entered. The temperature program defined should be that used for sample runs. No col umn compensation run is performed.
	Chosen detector (either A or B) not switched on. No column compensation run is performed.
	Chosen detector (either A or B) not present. No column compensation run IS performed.
	No detector(s) present. No column compensation run is performed.
	Occurs if entering new oven temperature program setpoints is attempted during a column compensation run. Entries are ignored. Also occurs if an attempt is made to start a column compensation run while one is already in progress. The one in progress continues to normal completion.

Typical Column Compensation Message Displays

A column compensation run terminates automatically at the completion of its oven temperature program. Any existing baseline profile is erased as data for the new baseline profile is collected and stored.

Note that the oven temperature program for a column compensation run follows setpoint values for initial time, rate, and final time as in an analytical run. Data is stored, however, only for rate and final time portions of the temperature program.

A sample run cannot be started via START while a column compensation run is in progress. Press: STOP to abort a column compensation run when the baseline profile stored is probably not valid (because the oven temperature program will not have reached the final temperature setpoint). A message WRONG TIME is displayed to indicate that a mismatch has occurred between the expected length of time for the run versus the actual time.

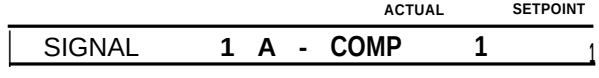
Assigning column compensation data

After baseline data for a given detector is stored as either COMP 1 or COMP 2, the column compensation data must be assigned to a specific detector signal. During a run, the compensation data is subtracted from run data for the same detector.

The following key sequence assigns such baseline-corrected data to a particular output channel:

Press: SIG 1 (or SIG 2) A (or B) − COL COMP1 (or COL COMP2) ENTER .

The figure below illustrates the display confirming the assignment:



Typical Display for Column Compensation

Note: No internal verification is given by the HP 5890 to ensure that compensation data collected on a given detector is assigned later to be subtracted from the same detector via the above key sequence. If you get strange baseline behavior from subtracting compensation data:

- Compensation data itself is suspected.
- Data acquired from a different detector has been assigned.
- Chromatographic conditions used for sample analyses are different from those used for the original column compensation run.

After you assign a particular output channel, sample analyses are performed in the usual manner, the only difference being that the observed baseline should be relatively free of drift.

Operating the nitrogen-phosphorus detector (NPD)

The nitrogen-phosphorus detector (NPD) uses a jet and collector similar to the FID. However, the collector contains a small alumina cylinder coated with a rubidium salt (the active element), which is heated electrically, creating a thermionicsource. In this environment, nitrogen and phosphorus containing organic molecules are ionized. The detector collects the ions and measures the resulting current.

As with an FID, an NPD requires hydrogen and air, but at lower flows. Therefore, normal FID-type ionizations are minimal, as is response to compounds not containing nitrogen or phosphorus. Thus, the detector is both sensitive to and selective of compounds containing nitrogen and/or phosphorus.

The electrical power for heating the active element is supplied through a toroidal transformer located inside the NPD cover. The toroidal transformer secondary winding is connected directly to the collector/active element assembly. The electrical heating current passes directly through the small platinum wire that is also used to position the active element inside the collector.

The active element of the NPD operates in a very delicate thermal balance that depends on several different variables. The magnitude of the response of the NPD is a function of the temperature of the active element and of the active zone around the active element itself. Because of this temperature dependence, the output of the detector is very sensitive to anything that affects the temperature of this active zone. Important variables and their effects include the following:

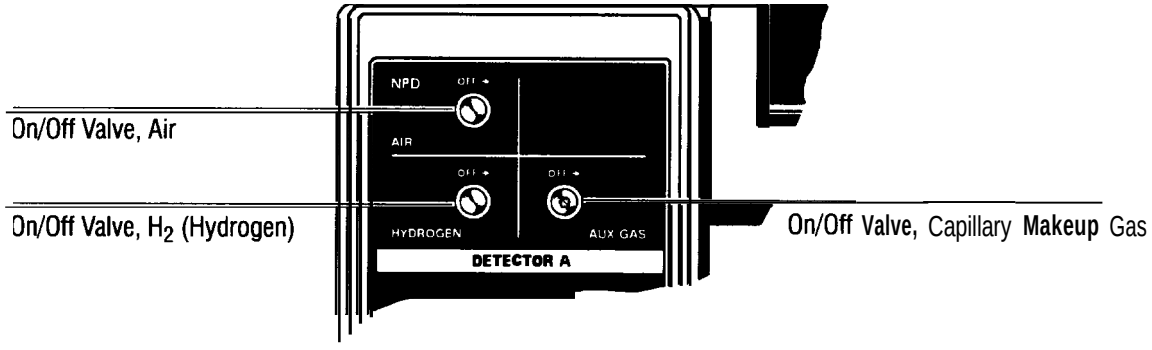
- Increasing detector temperature increases the active element temperature and the response.
- Increasing the electrical power to the active element increases both the temperature of the active element and the response.
- Increasing the hydrogen flow increases the temperature of the active element as well as the size of the active zone around the active element; both effects result in increased response.
- Increasing the air flow to the detector normally cools the active element slightly and decreases the response. (The change in temperature from altering the air flow is much less than the change from altering the hydrogen flow.) Increasing the air flow also decreases the residence time of a given peak in the active zone of the active element and decreases response.
- Increasing the carrier gas flow cools the active zone slightly and decreases the residence time of a component in the active zone, which decreases the response.

A hydrogen flow rate that is too high may cause a true flame around the active element. This would severely overheat the active element and destroy the specific response. An air flow rate that is too low may quench the background response of the active element, resulting in a reequilibration time that is too long to establish a proper background response (negative solvent peaks kill the active element).

Setting up the NPD for operation

To setup your NPD, you must do the following:

- Set the flow (for either packed or capillary columns).
- Condition the active element (bead).
- Set the active element power.
- Turn on the NPD.



Flow Panel Controlling NPD Operation

Conditioning the NPD active element (bead)

Condition the NPD active element (bead) when you install a new element or when the detector has been turned off for a period of time. Conditioning removes water that may have been absorbed into the active element from humidity in the air. If the active element is electrically heated too rapidly, the rubidium coating on the active element can fracture and ruin the collector.

This section assumes that the detector support gases are connected, the system is leak-free, the correct jet is installed, and a column is installed.

1. Set and turn on the carrier and detector gases.
2. Turn the active element power control fully counterclockwise to set the power to 000.

Note: The locking lever immediately below the control knob is locked in the right-most position and unlocked in the left-most position.

3. Turn off the power to the detector and the active element by pressing:

DET [A] (or [B]) [OFF]

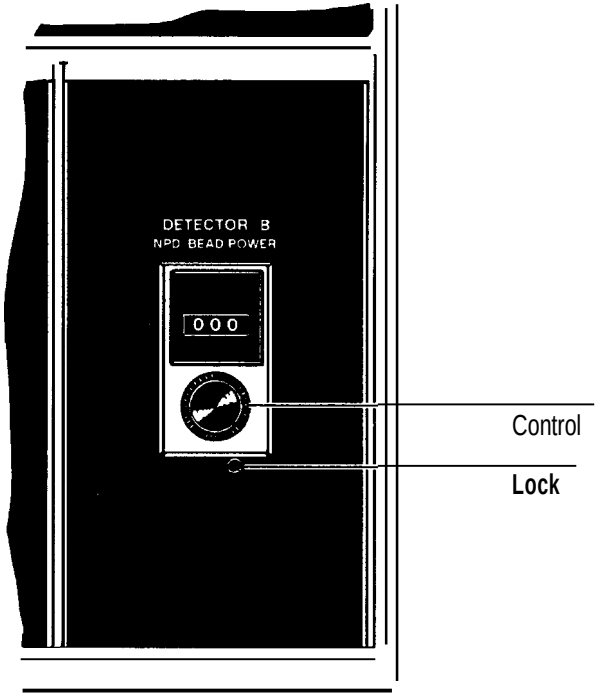
4. Set the oven temperature to 50°C

5. With the carrier and detector gases flowing, raise the temperature of the detector's isothermal zone to 220°C. Allow the collector to condition for at least 30 minutes under these conditions.

Note: If your detector is exposed to high humidity, condition new collectors for a longer period (overnight).

After the active element has been dried, initiate the specific NPD response by setting the power to the active element.

Setting the NPD active element (bead) power



NPD Active Element (Bead) Power Control

The following procedure sets the correct operating temperature for the active element. Operating at a temperature higher than the recommended range produces greater sensitivity at the expense of increased noise and reduced element lifetime with no increase in minimum detectable limit.

The temperature of the active element in the NPD collector is controlled by a 10-turn rotary control located below the keyboard. A mechanical counter (000 through 999) registers the position of the control.

Before setting the active element power, install a column and set the NPD flows as explained later in this section.

1. Set the heated zones to the desired operating temperatures. Leave the oven at 50°C

   Note: The temperature of the heated zone should be at least 200°C.

2. Turn off the power to the detector and the active element by pressing:

   DET A (or B) OFF

WARNING Do not leave the detector on while setting the active element power; it may overheat and become permanently damaged.

3. Set the active element power control to 000 by turning it fully counterclockwise.

   Note: The locking lever immediately below the control knob is locked in the right-most postion and unlocked in the left-most position.

4. Before setting the active element power, enter the following:

   DET A (or B) ON

   SIG 1 (or SIG 2) A (or B) ENTER

   SIG 1 (or SIG 2) *displayed value*

   The displayed value on the Oven/Det status window is in pA and should be close to zero. Allow approximately 1 minute for the baseline to stabilize.

5. Increase the element power slowly by turning the active element power control clockwise in steps of 100 (waiting 1 minute in between steps) until the displayed signal value approaches the target value. Expect little or no change at first, then a rapid increase as the element becomes active.

Generally the detector is adequately sensitive when enough power is supplied to the active element to display a signal output value in the range of 20–30 pA.

Note: Operational power settings vary depending on the hydrogen and air flow rate, the detector temperature, contamination, and the desired sensitivity For the conditions in this procedure, typical values range from 400–900. The power control can probably be increased immediately to 300–400 while giving a negligible increase in offset on the detector. As higher power settings are approached (500–600), increase the power more slowly and carefully

If you know the element power setting, bring power slowly to the setting and fine-tune if necessary. Wait for the baseline to stabilize before setting a final value.

After reaching the proper range of offset, allow the detector to stabilize before expecting precise measurements. The offset may drift until the active element becomes fully acclimated to the operating conditions. During this period, the apparent sensitivity of the detector will change.

Setting the NPD flow for packed columns

The gas flow rates outlined in this procedure ensure good, reliable detector behavior for the majority of analyses. If it is necessary to modify the flow rates for a specific application, use a standard sample matched to the application and experiment with the flow rates to optimize the detector's behavior.

WARNING To minimize the risk of explosion when using a bubble flow meter, never measure air and hydrogen together. measure them separately.

This section assumes that the detector support gases are connected, the system is leak-free, the correct jet is installed, and a column is installed.

1. Close the Aux gas on/off valve.
2. Attach a bubble flow meter to the NPD collector using the rubber adapter.
3. Set the column flow to approximately 20 ml/min.
4. Set the oven and the heated zones to the desired operating temperatures.
5. Set the column flow rate to 20 ml/min. Because the procedure for setting the column flow rate depends on the type of column installed and inlet system used, refer to the information about the appropriate inlet system in Chapter 4, *Setting Inlet System Flow Rates.*
6. Use the following steps to set the H_2 flow rate to 3 to 4 ml/min:
   a. Open the H_2 on/off valve by turning it counterclockwise. Measure the total flow rate (column plus makeup) through the detector.
   b. Adjust the H_2 pressure to the detector to obtain a total flow rate (H_2 plus column) of 23 to 24 ml/min.
   c. Turn the H_2 off.
7. Use the following steps to set the air flow rate to 100 to 120 ml/min:
   a. Open the air on/off valve by turning it counterclockwise. Measure the total flow
   (column plus air) through the detector.
   b. Adjust the air pressure to the detector to obtain a total flow of 120 to 140 ml/min.
   c. Turn the air flow off.
8. Remove the flow measuring adapter and bubble flow meter from the NPD collector.
9. Open the H_2 on/off valve.

Setting the NPD flow for capillary columns

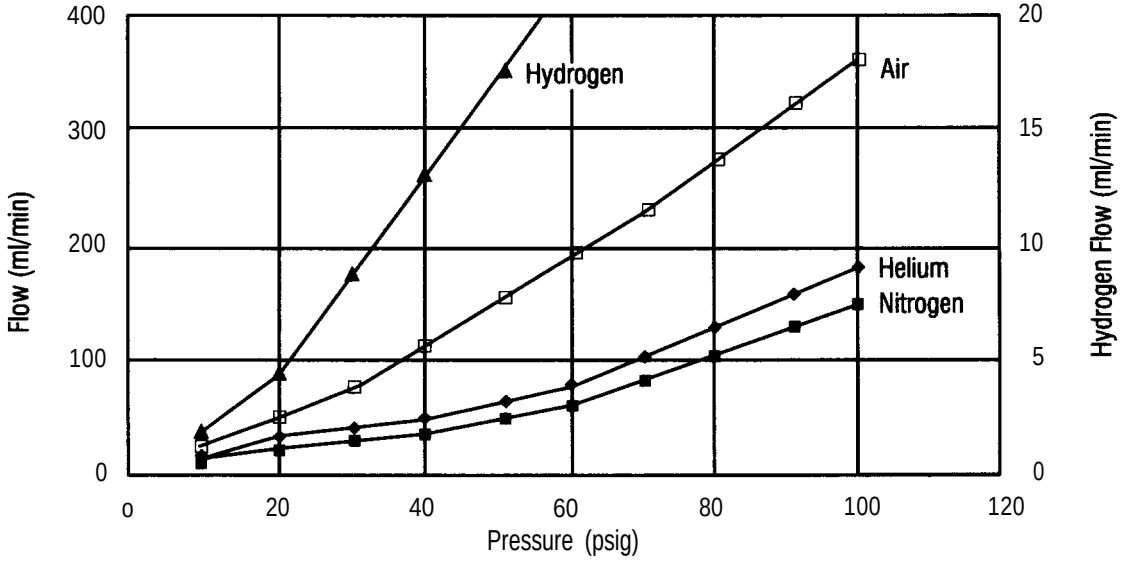
The table and graph on the following page show the pressure and flow values for EPC of the NPD.

Typical Pressure versus Flow for FID Flow Restrictors

Values computed using ambient temperature of 21°C and pressure of 14.56 psi

Flow Restrictor Pressure		FID Makeup HP pn 19243-60540 Green and Red Dots Flow (ml/min)		FID Hydrogen HP pn 19231-60660 Red Dot Flow (ml/min)	FID Air HP pn 19234-60600 Brown Dot Flow (ml/min)
kPa	psig	Nitrogen	Helium	Hydrogen	Air
69.0	10.0	6.4	7.2	2.0	21.0
137.9	**20.0**	15.0	17.0	4.6	45.0
206.8	**30.0**	26.0	29.0	8.1	76.0
275.8	**40.0**	39.0	44.0	12.4	110.0
344.7	**50.0**	53.0	61.0	17.0	148.0
413.7	**60.0**	69.0	80.0	22.3	188.0
482.6	**70.0**	86.0	100.0		229.0
551.6	**80.0**	104.0	122.0		273.0
620.5	**90.0**	123.0	150.0		318.0
689.5	100.0	143.0	178.0		363.0

= Recommended calibration points for using EPC with the HP 3365 ChemStation



NPD Restrictors

WARNING To minimize the risk of explosion when using a bubble flow meter, never measure air and hydrogen together. measure them separately.

This section assumes that the detector support gases are connected, the system is leak-free, the correct jet is installed, and a column is installed.

1. Set the oven and the heated zones to the desired operating temperatures.
2. Set the column flow rate to the desired value. Because the procedure for setting the column flow rate depends on the type of column installed and inlet system used, refer to the information about the appropriate inlet system in Chapter 4.
3. Adjust the total carrier and makeup gas flow (column plus makeup) to at least 30 ml/min.
4. Attach a bubble flow meter to the NPD collector using the rubber flow measuring adapter.
5. Use the following steps to set the capillary makeup gas flow rate for either manual or electronic pressure control.

Manual pressure control:

a. Set the supply pressure for the capillary makeup gas to about 276 kPa 40 psi).

b. Open the Aux gas on/off valve by turning it counterclockwise. Measure the total flow rate (column plus makeup) through the detector.

c. Use a small screwdriver to turn the variable restrictor at the center of the on/off valve to obtain a total flow rate of 30 ml/min (column plus makeup).

d. Use the following steps to set the H_2 flow rate to 3 to 4 ml/min:

i. Open the H_2 on/off valve by turning it counterclockwise. Measure the total flow rate (column plus makeup plus H_2) through the detector.

ii. Adjust the H_2 pressure to the detector until the total flow reaches 33 to 34 ml/min.

iii. Turn the H_2 flow off.

e. Use the following steps to set the air flow rate to 100 to 120 ml/min:

i. Open the air on/off valve by turning it counterclockwise. Measure the total flow rate (column plus makeup plus air) through the detector.

ii. Adjust the air pressure to the detector until it reaches 100– 120 ml/min.

Electronic pressure control:

a. Set the supply pressure to the auxiliary EPC channel to 40 psi.

b. Open the Aux gas (makeup gas) on/off valve. Turn the variable restrictor valve fully counterclockwise. Then adjust the pressure to get the desired flow rate.

c. Select the pressure units you would like to use.

To change the units, press: gold 1 ENTER . Then press the number of the corresponding unit you want to use:
1 = psi
2 = bar
3 = kPa

d. The example below sets the auxiliary channel C pressure to 10 psi.

Press:
gold A 1 0 ENTER Sets *auxiliary channel C pressure to* 10 psi.

	ACTUAL	SETPOINT
EPP C	10.0	10.0

The GC display looks like this

Note: To keep the pressure constant through an oven ramp program, see Chapter 10, *Using Electronic Pressure Control.*

e. Adjust the makeup gas pressure to the detector as necessary to obtain 30 ml/min total flow rate (column plus makeup).

6. Remove the bubble flow meter from the NPD collector.

7. Open the H_2 on/off valve.

Turning the NPD on and off

WARNING Do not leave the detector on while setting the active element power; it may overheat and be permanently damaged.

To turn the NPD on, press: DET A (or B) ON.

To turn the NPD off, press: DET A (or B) OFF.

After the oven and zones reach the desired setpoint values, wait an additional ½hour before using the NPD.

Optimizing the performance of the NPD

To optimize the performance of the NPD, you should avoid contamination of the detector and preserve the lifetime of the active element (bead).

Avoiding contamination

The slightest contamination can create serious NPD problems. The following list describes common sources of contamination to avoid:

- Columns and/or glass wool treated with H_3PO_4 (phosphoric acid)
- Phosphate-containing detergents
- Cyano-substituted silicone column (such as XE-60 and OV-225)
- Nitrogen-containing liquid phases
- Any liquid phase deactivated for analysis of basic compounds
- Fingerprints
- Leak-detection fluids
- Laboratory air

Contamination may affect the performance of an NPD in two ways:

- Positive contamination gives a more positive offset than what would normally result from a clean system. In response to the positive offset, you may operate the detector with too little power to the active element. Because the temperature of the active element is less than normal, the detector appears less sensitive than is desirable.
- Negative contamination quenches the reaction, resulting in decreased sensitivity. Very high contamination may completely quench all signals from the detector. If this happens, the apex of a peak is flattened toward the baseline.

Preserving the lifetime of the active element

The lifetime of the active element is reduced by silicon dioxide coating, loss of rubidium salt, and humidity. Observe the following suggestions to preserve the lifetime of the active element:

- Prevent silicon dioxide from coating the active element. Residual silanizing reagents from derivatization, and/or bleed from silicone columns, may coat the active element with silicon dioxide. This decreases ionization efficiency reducing sensitivity.

  If silanizing is necessary, remove excess reagent before injection. Silicone columns should be well conditioned and loaded less than 5%.

- Do not overheat the active element. Rubidium loss is caused by overheating the active element, particularly if the element power is on when gas flows are interrupted (especially in the carrier). You must turn off the detector or reduce the element power to zero when changing the columns and/or replacing the gas cylinders. Power to the element while the gas flow is off can destroy an element within a few minutes.

- Use the lowest element power possible, consistent with maintaining sufficient detector sensitivity and selectivity for the particular analyses.

- Reduce the power to the active element whenever the detector will not be operated for extended periods of time (such as over the weekend).

To determine the proper amount of power reduction, plot the normal offset and note the displayed zero value (20–30 is in the normal range). Then reduce the power setting slightly until the displayed zero value (offset) just goes to zero or to a value close to zero (lower than 5 picoamps). In this way, the temperature of the active element will be lowered such that there will be little loss of rubidium, but the active element will still be kept hot enough to prevent contamination (condensation) while in standby.

- If you are using the auxiliary EPC channel to control the NPD gases, you can program hydrogen to a lower value. This cools the bead and thereby extends the bead life.
- Counteract humidity. Humidity adversely affects the lifetime of an element. Keep the detector warm (l00°C to 150°C) when it is not in use. Store the collector (including spare collectors) in a desiccator whenever you remove it from the NPD for an extended period of time.
- Recoat or replace an old element. Invest in a recoating kit, which rejuvenates the active element in an old collector. Also keep a spare collector available as a replacement.
- Generally sensitivity and selectivity to nitrogen decreases as the element ages. Phosphorus response is affected less than nitrogen response.
- Do not remove the seals that cover the NPD during shipments until you are ready to connect the column and operate the detector. Without the seals, the active element may become contaminated, which will reduce the collector's effectiveness and possibly ruin the active element.

Both the detector baseline and sensitivity change with the carrier flow rate due to changes in the temperature of the active element. This causes baseline drift in pressure-controlled inlet systems (capillary inlets) while temperature-programming the column. The amount of change in the detector response is proportional to the ratio of the total column flow change (temperature sensitive) to the makeup gas flow (not temperature sensitive); that is, total column flow change divided by makeup gas flow. Adjust the element power after any change in the carrier flow rate.

When the detector is first turned on, its sensitivity and signal level change slowly over several hours. Therefore, for applications requiring very stable operation, leave the detector on overnight, lowering the oven temperature to prevent contaminating the active element with column bleed.

Operating the electron capture detector (ECD)

This section explains how to operate an electron capture detector (ECD). Specifically, it describes the following:

- The basic operating characteristics of an ECD
- General issues to consider when using an ECD, including temperature, gases, flow rates, and background
- Routine detector operating procedures, including setting the column flow, setting the carrier gas selection switch, setting carrier gas and makeup gas flow rates, and performing daily startup and shutdown procedures.

WARNING The gas stream from the detector must be vented to a fume hood to prevent possible contamination of the laboratory with radioactive material. For cleaning procedures, see *Cleaning the detector* in this chapter.

Requirements for USA owners

WARNING Detector venting must be in conformance with the latest revision of Title 10, Code of Federal Regulations, Part 20 (including Appendix B).

The detector is sold under general license: owners may not open the detector cell or use solvents to clean it. Additional information is available in the publication information for general licensees, Pub. No. 43-5953-1586 (D).

Owners of this detector must perform a radioactive leak test (wipe test) at least every 6 months. See *Testing for radioactive leaks (the wipe test)* in this chapter.

WARNING

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

- After turning off the main power and allowing the instrument to cool, cap the ECD inlet and exhaust vent openings. Wear disposable plastic gloves and observe normal safety precautions.

Ž Return the cell for exchange following the directions included with the form general license certification (HP Pub. No. 43-5954-7621, HP part number 19233-90750).

Even in this very unusual situation, radioactive material is unlikely to escape the cell. Permanent damage to the ^{63}Ni plating within the cell is possible, however, so the cell must be returned for exchange.

Introduction

The electron capture detector (ECD) cell contains ^{63}Ni, a radioactive isotope emitting high-energy electrons (ß-particles). These undergo repeated collisions with carrier gas molecules, producing about 100 secondary electrons for each initial ß-particle.

Further collisions reduce the energy of these electrons into the thermal range. These low-energy electrons are then captured by suitable sample molecules, thus reducing the total electron population within the cell.

Uncaptured electrons are collected periodically by applying short-term voltage pulses to the cell electrodes. This cell current is measured and compared to a reference current. The pulse interval is then adjusted to maintain constant cell current.

Therefore pulse rate (frequency) rises when an electron-capturing compound passes through the cell. The pulse rate is converted to a voltage, which is related to the amount of electron-capturing material in the cell.

The ECD responds to compounds having an affinity for electrons—for example, halogenated materials such as pesticides and related compounds.

The following table shows expected sensitivities to different classes of organic compounds.

General considerations

General ECD Sensitivity to Various Classes of Compounds

Chemical Type	Relative Sensitivity
Hydrocarbons	1
Ethers, esters	**10**
Aliphatic alcohols, ketones, amines; mono-Cl, mono-F compounds	10^2
Mono-Br, di-Cl and di-F compounds	10^3
Anhydrides and tri-Cl compounds	10^4
Mono-1, di-Br and nitro compounds	10^5
Di-I, tri-Br, poly-Cl and poly-F compounds	10^6

The figures in the table are only approximate, and sensitivity varies widely within each group, depending on the structure of the material. For example, DDT with 5 chlorine atoms per molecule can be measured in the 1-to 10-picogram range.

Temperature effects

Some compounds exhibit strong response to detector temperature. The effect may be either positive or negative. Try different detector temperatures above the oven temperature to determine the effect on sensitivity Generally, a detector temperature between 250 and 300ºC is satisfactory for most applications.

Gases

The ECD is designed for use with either nitrogen or argon-methane as carrier gas. Nitrogen yields somewhat higher sensitivity with approximately the same minimum detectable limit, but is also accompanied by higher noise and occasional negative solvent peaks. Argon-methane gives a greater dynamic range. Use the appropriate switch to select carrier gas type. The ECD does not operate properly if the switch is set incorrectly.

Because of its high sensitivity never use the ECD without moisture, chemical, and 0_2 traps in carrier and makeup lines. The traps should be in good condition and installed in the carrier gas supply line and the makeup gas supply. Also, avoid using plastic tubing, which is permeable to most gases, for all connections. Use clean copper tubing instead.

Columns and flow rates

An ECD is normally used to detect compounds that are reactive enough to interact with metal columns. Therefore, only l/4-inch packed glass or fused silica capillary columns are recommended with this detector.

Hydrogen carrier gas (with nitrogen makeup gas) gives the best column performance.
Argon-methane can also be used as makeup gas. For most purposes, 50-60 ml/min of makeup gas is satisfactory, but the rate maybe increased to 100 ml/min for very fast runs. Because the ECD is a concentration-dependent detector, increasing the flow rate reduces sensitivity but can extend the linear range.

Note: When measuring ECD flow rates, attach a bubble flow meter directly to the detector exhaust vent using a small piece of rubber tubing as an adapter.

Background

If the ECD becomes contaminated from impurities in the carrier (or makeup) gas or from column bleed, a significant fraction of detector dynamic range may be lost. In addition, the output signal becomes noisy.

To check the background level, allow ample time for the components from the previous analyses to be flushed from the system and then make a blank run (one with no sample injected).

Setting up the ECD for operation

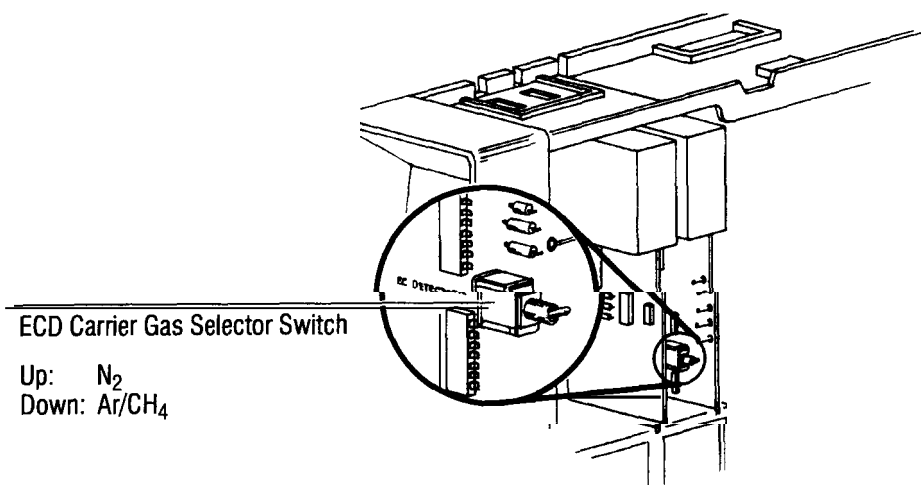
To setup the ECD for operation, you must:

- Set the carrier gas selection switch (if it is not done already).
- Set the ECD flow (for either packed or capillary columns).

Setting the carrier/makeup gas selection switch

The carrier gas selection switch is located on the detector board behind the right instrument side panel. Use the following procedure to set the carrier gas selection switch if necessary:

1. Turn off the power and unplug the instrument.
2. Remove the top right cover by lifting first at its rear edge and then sliding it toward the rear of the instrument.
3. Remove the right side panel by removing four screws, two along its bottom edge and two along its top edge.
4. Locate the ECD signal board, which is next to the detector.
5. Locate the N_2-ArCH_4 switch and place it in the appropriate position based on the type of predominant gas at the detector (carrier or makeup).
6. Replace the panels.



ECD Carrier Gas Selector Switch

Setting the ECD flow for packed columns

Gas flow rates given in this section ensure good, reliable detector behavior for most applications. To optimize detector behavior for a specific application, use a standard sample matched to the application and experiment with other flow rates.

Use the steps in the following procedure to set the ECD flow in the column. This procedure assumes that the detector support gases are connected, the system is leak-free, and a column is installed. If your system has electronic pressure control, enter the flows at the keyboard using the *Electronic pressure control* instructions.

1. Set the column flow to the desired rate. Because the procedure for setting the column flow rate depends on the column installed and the inlet system in use, refer to the appropriate system information in Chapter 4.

2. Open the ECD Anode Purge On/Off valve. Supply pressure of 30 psi will deliver approximately 3 ml/min of purge flow. ECD anode purge flow is not considered part of total column flow.

Packed column considerations: Either N_2 or Ar containing 5% CH_4 may be used as carrier gas. N_2 yields somewhat higher sensitivity, but it is accompanied by higher noise; minimum detectable limit is about the same. N_2 sometimes produces a negative solvent peak. Ar/CH_4 gives greater dynamic range.

Total flow of 60 ml/min is adequate in most applications to prevent peak broadening and maximize linearity.

The carrier gas must be dry and 02-free. Moisture and 0_2 traps are strongly recommended for highest sensitivity. Because plastic tubing is permeable to many gases, the use of clean copper tubing is recommended for all connections.

Setting the **ECD** flow for capillary columns

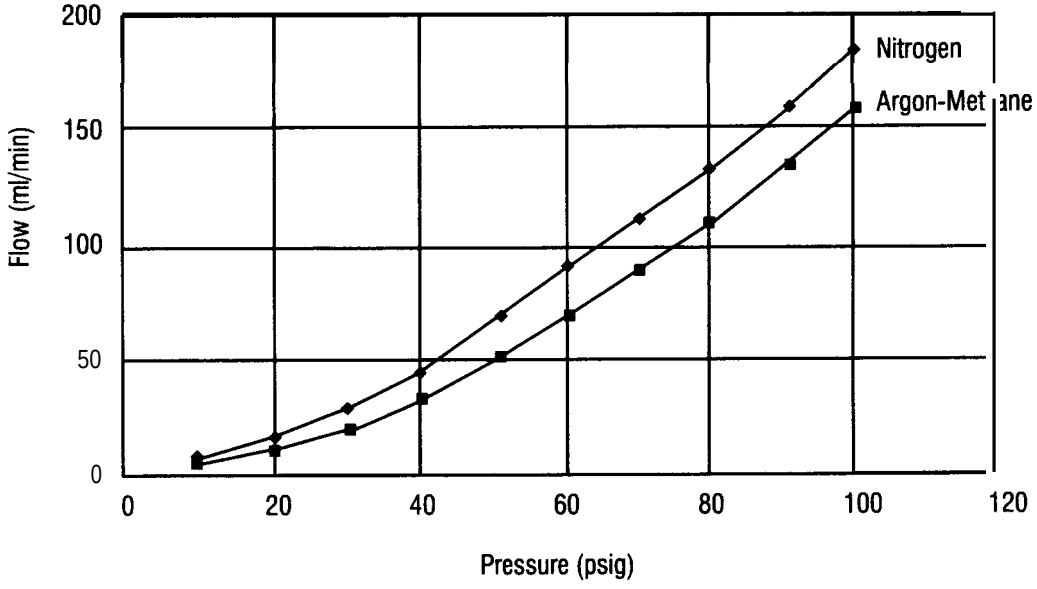
The following table and graph show the optimal pressure and flow values for EPC or the ECD.

Typical Pressure versus Flow for ECD Flow Restrictors
Values computed using ambient temperature of 21°C and pressure of 14.56 psi

Flow Restrictor Pressure		ECD Makeup HP pn 19231-60770 Red Dots Flow (ml/min)	
kPa	psig	Nitrogen	Argon-Methane
69.0	10.0	8.0	7.0
137.9	20.0	20.0	17.0
206.8	30.0	34.0	29.0
275.8	40.0	51.0	44.0
344.7	50.0	69.0	60.0
413.7	60.0	89.0	78.0
482.6	70.0	110.0	97.0
551.6	80.0	132.0	117.0
620.5	90.0	156.0	138.0
689.5	100.0	181.0	160.0

= Recommended calibration points for using EPC with the HP 3365 ChemStation

Note: The anode purge flow rates will be approximately l/10th of the values shown above at the same pressure settings.



ECD Restrictors

To optimize detector behavior for a specific application, use a standard sample matched to the application and experiment with other flow rates.

Use the steps in the following procedure to set the ECD flow in the column.

1. Set the column flow to the desired rate. Because the procedure for setting column flow rate depends on the column installed and the inlet system used, refer to the appropriate system information in Chapter 4.

   Supply pressure for the makeup gas and anode purge should be set to 207 kPa (30 psi).

2. Open (counterclockwise) the On/Off valve for ECD makeup gas flow. Supply pressure of 60 psi will deliver approximately 60 ml/min of makeup gas flow.

3. Open the ECD Anode Purge On/Off valve. Supply pressure of 60 psi will deliver approximately 6 ml/min of purge flow. ECD anode purge flow is considered part of total column flow.

Capillary column considerations: H_2 or He carrier gas affords the best column performance with reduced retention times. Ar/CH_4 or N_2 as makeup gas is used in the range of 60 ml/min. Because the ECD is a concentration-dependent detector, reduced sensitivity is obtained at higher flow rates.

For the ECD, capillary makeup gas should be used even with HP Series 530 µ capillary columns because the detector requires a total flow rate of at least 25 ml/min.

Moisture and 0_2 traps for carrier and makeup gas are essential with capillary/ECD operation.

Your ECD makeup gas is equipped with either manual or electronic pressure control. Set the makeup gas according to the instructions for your instrument.

Manual pressure control:

a. Set the supply pressure for the capillary makeup gas to about 276 kPa (40 psi).

b. Open the on/off valve for the ECD makeup gas flow by turning it counterclockwise.

c. Use a small screwdriver to turn the variable restrictor at the center of the on/off valve as necessary to obtain a flow of 60 ml/min.

Electronic pressure control:

a. Set the supply pressure to the auxiliary EPC channel to 40 psi.

b. Open the Aux gas (makeup gas) on/off valve. You will use the auxiliary EPC pressure to control the auxiliary gas flow rate.

c. Select the pressure units you would like to use.

To change the units, press: gold 1 ENTER . Then press the number of the corresponding unit you want to use:

1 = psi
2 = bar
3 = kPa

d. With electronic pressure control, makeup gas is controlled through auxiliary pressure channels C, D, E, or F from the keyboard. The example below sets the auxiliary channel C pressure to 40 psi.

Press:

gold A 4 0 ENTER *Sets auxiliary channel C pressure to 10 psi*

	ACTUAL	SETPOINT
EPP C	40.0	40.0

The GC display looks like this

Note: To keep the pressure constant through an oven ramp program, see Chapter 10, *Using Electronic Pressure Control.*

e. Adjust the makeup gas pressure to the detector as necessary to obtain an appropriate total flow rate (column plus makeup).

For the ECD, use capillary makeup gas even with HP Series 530 µ capillary columns because the large cell size requires high total flow rate (at least 50–60 ml/min).

For an ECD, the makeup gas is added into the column effluent stream via a capillary makeup gas adapter fitted into the detector column inlet.

Testing for contamination

Because of its very high sensitivity the ECD is particularly prone to contamination problems, including contaminants entering the system via the carrier and makeup gas source.

Perform the following procedure whenever a new carrier gas source is installed:

1. With the instrument on and operating normally, cool the oven to ambient temperature, turn off the detector, turn off carrier flow to the detector, and remove the column to the ECD. If a capillary column was installed, also remove the makeup gas adapter in the detector base.

2. Disconnect the carrier gas source line at its fitting on the rear of the inlet used.

   Note: If your carrier gas is helium or hydrogen (not N_2 or Ar-CH_4), then use the makeup gas, not the carrier gas.

3. Using a Vespel ferrule, and adapters as necessary, connect the carrier source line to the detector base, including any traps in the line.

4. Set the carrier pressure to about 7 kPa (1 psi) and check for flow through the detector.

5. Leaving the oven door open, enter any temperature for the detector up to 250°C.

6. Turn on the detector electronics.

7. Assign the ECD to one of the monitored signals.

8. Within 15 minutes, the displayed signal values on the Oven\Det Status window should be between 40 to 100 (400 to 1,000 Hz); there may be downward drift.

9. If the displayed values are greater than 1,000 Hz, the trap(s) maybe at fault. Connect the carrier gas supply line directly to the detector base and repeat the test. If the values are still out of range, then the carrier gas supply or the detector maybe contaminated. Try a new tank of N_2 or Ar-CH_4. If the system is fine, then the gas was contaminated. If not, the cell is probably dirty and should be exchanged.

Testing for leaks

Note: This test assumes that the flow system components upstream from the detector are leak-free.

Use the steps in the following procedure to test for leaks at the ECD:

1. Set the inlet, oven, and detector to ambient temperature and allow time for cooling. Turn off the detector and carrier flow.
2. Use a vent plug to cap the ECD exhaust vent.
3. Set the carrier gas pressure to an appropriate valve depending on the inlet system you are using. Open the carrier gas mass flow controller fully to ensure that flow through the system is available. Allow time for the system to become fully pressurized.
4. Close the carrier gas flow at its source and monitor system pressure.
5. If no pressure drop is observed over a 10-minute period, assume that the system is leak-free.
6. If leakage is observed, use an appropriate electronic leak detector to check for leaks at the detector column fittings and at the plugged vent.

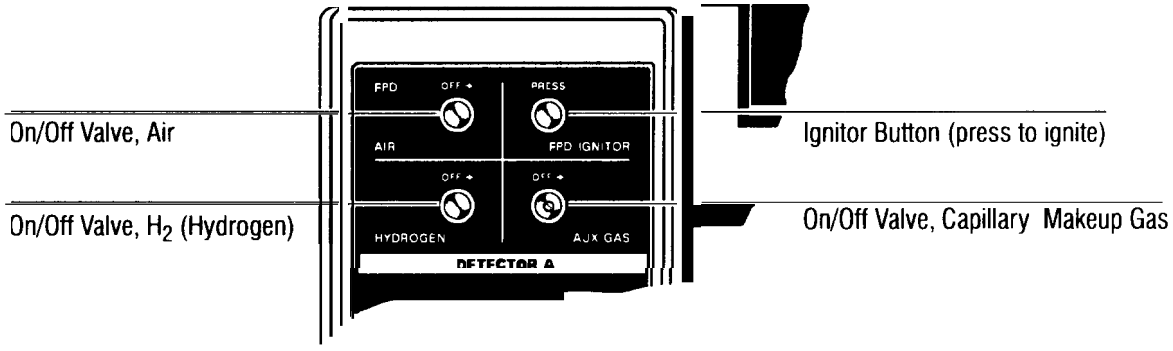
   Note: The detector body itself is not a likely source of leaks. It cannot be disassembled without special license from the Nuclear Regulatory Commission or Agreement State Licensing Agency (USA only).

Testing for radioactive leaks (the wipe test)

ECDs must be tested for radioactive leaks at least every 6 months. Records of tests and results must be maintained for possible inspection by the Nuclear Regulatory Commission and/or responsible state agency. More frequent tests may be conducted when necessary

A wipe test kit, supplied with each new ECD, contains complete instructions for conducting the test.

Operating the flame photometric detector (FPD)



Flow Panel for Controlling FPD Operation

Setting up the FPD for operation

To setup the FPD for operation, you must:

- Set the FPD flow (for either packed or capillary columns).
- Set the FPD sensitivity.
- Turn on the FPD.

Setting the **FPD** flow for packed columns

The gas flow rates given in this section ensure good, reliable detector behavior for most applications. To optimize detector behavior for a specific application, use a standard sample matched to the application and experiment with other flow rates.

WARNING Flame photometric detectors use hydrogen gas as fuel. if hydrogen flow is on and no column is connected to the detector inlet fitting, hydrogen gas can flow into the oven and create an explosion hazard. Inlet fittings must have either a column or a cap connected whenever hydrogen is supplied to the instrument.

Use the steps in the following procedure to set the FPD flow in a packed column. This procedure assumes that detector support gases are connected, the system is leak-free, and a column is installed.

1. Close the Aux gas on/off valve.
2. Set the column flow rate to 20 ml/min. Because the procedure for setting column flow rate depends on the column installed and the inlet system used, refer to the appropriate inlet system information in Chapter 4.
3. Set the oven and heated zones to the desired operating temperatures.
4. Attach a bubble flow meter to the FPD vent tube.

WARNING To minimize risk of explosion when using a bubble flow meter, never measure air and hydrogen together. Measure them separately.

To optimize sulfur sensitivity use a lower hydrogen flow rate (50–60 ml/minis recommended). To optimize phosphorus sensitivity use a higher hydrogen flow rate (about 100 ml/min is recommended).

5. Use the following steps to set the H_2 flow rate to 75 ml/min:

   a. Open the H_2 on/off valve by turning it counterclockwise. Measure the total flow rate (column plus H_2) through the detector.

   b. Adjust the H_2 pressure to the detector to obtain a total flow rate (column plus H_2) of about 95 ml/min.

   c. Close the H_2 on/off valve.

6. Use the following steps to set the air flow rate to 100 ml/min.

   a. Open the air on/off valve by turning it counterclockwise and measure the total flow rate (column plus air) through the detector.

   b. Adjust the air pressure to the detector to obtain a total flow rate (column plus air) to 120 ml/min.

7. Remove the measuring adapter from the FPD collector.

8. Open the H_2 on/off valve. To ignite the flame, see *Igniting the FPD flame* later in this chapter.

Setting the FPD flow for capillary columns

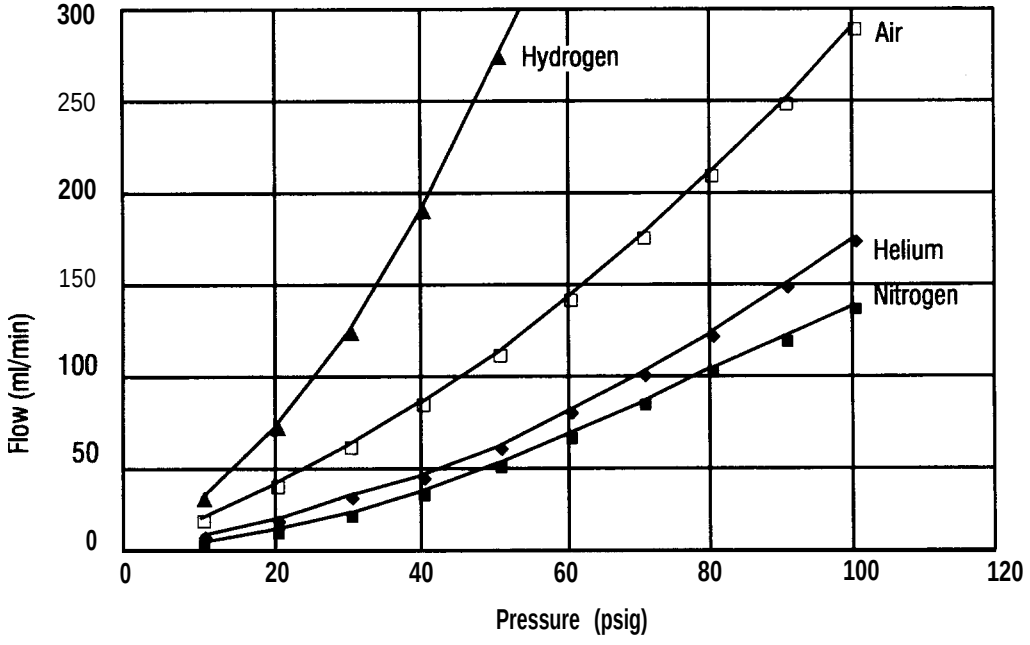
This table and graph show the optimal flow rates at which to control your FPD with EPC.

Typical Pressure versus Flow for FPD Flow Restrictors

Values computed using ambient temperature of 21°C and pressure of 14.56 psi

Flow Restrictor Pressure		FPD Makeup HP pn 19243-60540 Green and Red Dots Flow (ml/min)		FPD Hydrogen HP pn 19234-60570 Red Dot Flow (ml/min)	FPD Air HP pn 19234-60570 Brown Dot Flow (ml/min)
kPa	**psig**	**Nitrogen**	**Helium**	**Hydrogen**	**Air**
69.0	10.0	6.4	7.2	31.0	15.0
137.9	20.0	15.0	17.0	73.0	34.0
206.8	30.0	26.0	29.0	126.0	57.0
275.8	40.0	39.0	44.0	189.0	84.0
344.7	50.0	53.0	61.0	259.0	113.0
413.7	60.0	69.0	80.0	336.0	143.0
482.6	70.0	86.0	100.0		178.0
551.6	80.0	104.0	122.0		212.0
620.5	90.0	123.0	150.0		248.0
689.5	100.0	143.0	178.0		284.0

= Recommended calibration points for using EPC with the HP 3365 ChemStation



FPD Restrictors

The gas flow rates given in this section ensure good, reliable detector behavior for most applications. To optimize detector behavior for a specific application, use a standard sample matched to the application and experiment with other flow rates.

WARNING

Flame photometric detectors use hydrogen gas as fuel. If hydrogen flow is on and no column is connected to the detector inlet fitting, hydrogen gas can flow into the oven and create an explosion hazard. Inlet fittings must have either a column or a cap connected whenever hydrogen is supplied to the instrument.

Use the steps in the following procedure to set the FPD flow in a capillary column. This procedure assumes that the detector support gases are connected, the system is leak-free, and a column is installed.

1. Set the oven and the heated zones to the desired operating temperatures.
2. Set the column flow to the desired rate. Because the procedure for setting column flow rate depends on the column installed and the inlet system used, refer to the appropriate inlet system information in Chapter 4.
3. Set the carrier and makeup gas flow rate (column plus makeup) through the detector to at least 20 ml/min.
4. Attach a bubble flow meter to the FPD vent tube.

   Your FPD makeup gas is equipped with either manual or electronic pressure control. Set the makeup gas according to the instructions below. To set capillary makeup gas flow rate:

   Manual pressure control:

   a. Set the supply pressure for the capillary makeup gas to about 276 kPa (40 psi).

   b. Open the Aux gas on/off valve for the FPD makeup gas flow by turning it counterclockwise.

c. Use a small screwdriver to turn the variable restrictor at the center of the Aux gas on/off valve as necessary to obtain 20 ml/min total flow rate (column plus makeup).

Electronic pressure control:

a. Set the supply pressure to the auxiliary EPC channel to 60–70 psi using the keyboard.

b. Open the Aux gas (makeup gas) on/off valve. Use the auxiliary EPC pressure to control the auxiliary gas flow rate.

c. Select the pressure units you would like to use.

To change the units, press: gold 1 ENTER. Then press the number of the corresponding unit you want to use:

1 = psi
2 = bar
3 = kPa

d. The example below sets the auxiliary channel C pressure to 10 psi.

Press:

gold A 1 0 ENTER *Sets auxiliary channel C pressure to 10 psi*

	ACTUAL	SETPOINT
EPP C	10.0	10.0

The GC display looks like this

Note: To keep the pressure constant through an oven ramp program, see Chapter 10, *Using Electronic Pressure Control.*

e. Adjust the makeup gas pressure to the detector as necessary to obtain 20 ml/min total flow rate (column plus makeup).

WARNING To minimize risk of explosion when using a bubble flow meter, never measure air and hydrogen together. Measure them separately.

5. Set the H_2 flow rate to 75 ml/min. If you have a manually controlled system, use the steps below. If you have EPC, enter the flow rate from the keyboard as you did for makeup gas.

a. Open the H_2 on/off valve by turning it counterclockwise. Measure the total flow rate (column plus makeup plus detector) through the detector.

b. Adjust the H_2 pressure to the detector to obtain a total flow rate (column plus makeup plus H_2) of about 95 ml/min.

c. Close the H_2 on/off valve.

6. Use the following steps to set the air flow rate to 100 ml/min:

a. Open the air on/off valve by turning it counterclockwise and measure the total flow rate (column plus makeup plus air) through the detector.

b. Adjust the air pressure to the detector to obtain a total flow rate (column plus makeup plus air) to 120 ml/min.

7. Remove the flow measuring adapter from the FPD collector.

8. Open the H_2 on/off valve.

Turning the FPD on and off

After setting the flows, you can turn the FPD on or off.

To turn the FPD on, press: DET A (or B) ON.

To turn the FPD off, press: DET A (or B) OFF.

Igniting the FPD flame

WARNING To minimize the risk of explosion, do not attempt to ignite the FPD flame by applying a flame at its exhaust tube. Follow the procedure below.

Note: After the flows are set, the FPD flame is relatively easy to ignite. The detector module is most easily lit if heated to at least 200°C. Use the sequence described below to avoid a loud pop on ignition.

1. Turn all FPD flows (except the column flow) off
2. If required, open the auxiliary N_2 valve.
3. Open the air (or 0_2) valve.
4. Press in and hold the igniter button.
5. Open the H_2 valve.

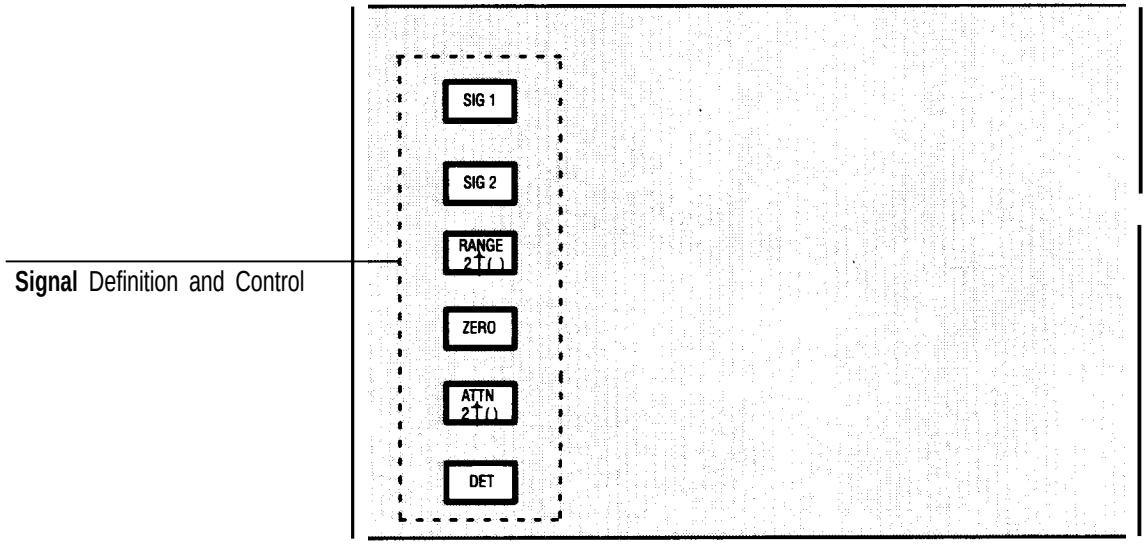
   Note: Always open the hydrogen valve after opening the air (or 0_2) and pressing the igniter. Failure to do this will result in a loud pop. This should not damage the detector, but is unpleasant to hear.

6. Release the igniter button.

   Proper ignition should result in a slightly audible pop. Flame ignition can be verified by holding a mirror or a cold, shiny surface near the exhaust tube and observing condensation. Ignition also usually results in a small increase in signal offset on the LED display.

Controlling Signal Output

Controlling Signal Output



Output sources include detector signal(s), heated zone or oven temperatures, carrier gas flow rates, column compensation run data, or test chromatographic data. If both signal channels are present, each may output information simultaneously from the same source, or from two different sources.

Each channel provides two levels of analog output:

0 to +1 mV:	for strip chart recorders.
-0.01 to +1 V:	for electronic integrators with analog inputs.

The two output levels are independent and may be connected simultaneously to separate data-receiving devices.

Note: A **tick** mark (electrical pulse) is produced at the **+1 mV** analog output when either START or STOP is pressed and when a run times out (run time elapses). These marks locate beginning and ending points in a chromatogram plotted at a continuously running strip chart recorder.

Assigning a signal

After the appropriate signal channel is displayed by pressing SIG 1 (or SIG 2, if Option 550/Accessory 19242A or Option 560/Accessory 19254A is installed), any one of the instrument functions in the following table may be entered to assign the signal to be output from the displayed channel.

Note that in a two-channel instrument it is permissible to have the same signal assigned to each signal channel, allowing identical data to be treated differently simultaneously.

Key(s)	Notes
A or B	To output the signal from either detector **A** or detector **B** The message DET A (or B) NOT INSTALLED is displayed if detector **A** (or **B)** is not present.
A – B or B – A	To output a difference signal between two detectors of the same type The message DET A (or B) NOT INSTALLED is displayed if detector **A** (or **B)** is not present; the message UNLIKE DETECTORS is displayed if detectors **A** and **B** are not of the same type.
A – COL COMP1 or B – COL COMP1 or A – COL COMP2 or B – COL COMP2	To output a difference signal between a given detector and column and a stored **blank run** signal for the detector and column The message DET A (or B) NOT INSTALLED is displayed if detector **A** (or **B)** is not present.
0	To output oven temperature
1 or 2	To output stored **COMP 1** or **COMP 2** data
3 , 4 , 5 or 6	To output, respectively, inlet **A** temperature, inlet **B** temperature, detector **A** temperature, or detector **B** temperature
7 or 8	To output, respectively, carrier gas flow rate **A** or **B**
9	To output a test signal (stored chromatogram) for use in verifying proper operation of a data-receiving device (integrator, chart recorder, etc); details are discussed later in this section.

As an example, a key sequence to assign detector **B** data to the **Signal 1** output channel would be:

SIG 1 B ENTER

At the same time, **A** flow rate data (if electronic sensing is installed could be assigned to the **Signal 2** output channel:

SIG 2 7 ENTER

As an assignment is made for each channel, confirmation is given through appropriate displays.

Displaying or monitoring a signal

By pressing the appropriate signal key (SIG 1 or SIG 2), current status of the corresponding signal channel is displayed.

Two types of displays are possible: either a display showing the instrument function assigned to the particular signal channel or a display monitoring the current actual output value for the assigned instrument function. Repeatedly pressing SIG 1 (or SIG 2) switches between the two possible display types.

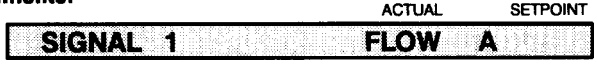
Detector Signal Assignments:

Detector Signal Monitoring:

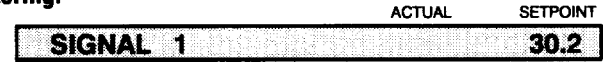
Oven/Zone Temperature Assignment:

Oven/Zone Temperature Monitoring:

Flow Rate Assignments:



Flow Rate Monitoring:



Typical Signal Displays:

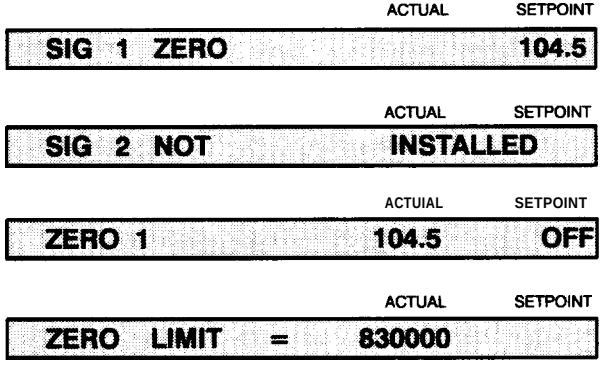
Signal monitoring is useful, for example, in determining if an FID is ignited, in setting active element current for an NPD, in determining cleanliness of an ECD, in tracking temperatures or gas flow rates, etc. The monitored value displayed is unaffected by scaling functions performed by ZERO, RANGE 2↑(), and/or ATTN 2↑() (these key functions are discussed later).

If oven or heated zone temperature is monitored via the display the conversion factor between the displayed value versus actual temperature is 64 counts/°C –200. Similarly if flow rate is monitored via the display, the conversion factor is 32 counts/(ml/min).

Zeroing signal output

When using analog signal output, using [ZERO] can increase dynamic range available by subtracting a constant background signal from the detector signal. Background signal sources include the detector itself (background level depending upon detector type), column bleed, or contaminants in supply gas(es). There are limits to this, however, so it is good practice to reduce background as much as possible by minimizing column bleed by using clean supply gases and by performing proper detector maintenance.

Current [ZERO] setpoint value is displayed by pressing the appropriate signal channel key ([SIG 1] or [SIG 2]), followed by [ZERO] (or simply press [ZERO] alone, if the desired signal channel is already displayed). Typical displays are shown below.



Typical Zero Displays

Once current [ZERO] setpoint value is displayed for the desired signal channel, pressing [ENTER] causes the value to be changed to the current signal value.

Zeroing should be done only at times of quiet chromatographic activity **(i.e., not** during a run). To do so during an active run may cause a baseline shift at the recording/integrating device.

If the [ZERO] setpoint value determination is not appropriate for a particular application, any value from –830000.0 through 830000.0 may be entered at the keyboard.

Entering a value **less** than the current [ZERO] value shifts background baseline **upward** (but at the expense of available output range); for example, to capture negative peaks or to compensate for negative baseline drift.

Turning zero off/on

When the current setpoint offset value for [ZERO] is displayed for the desired output channel, pressing [OFF] halts subtracting the offset value from the signal. Baseline is restored to its absolute level with respect to the HP 5890 electrical zero.

- The current setpoint value remains stored; however, pressing [ON] resumes subtracting the offset value from the signal.
- If [ZERO] is off, pressing [ENTER] switches the zeroing function on and causes a [ZERO] determination.

Setting signal attenuation

For analytical information from a detector, [RANGE 2↑()] and [ATTN 2↑()] are used to keep peaks of interest on scale at the integrator or chart recorder. Peaks of interest must neither **flat top** by exceeding the allowed maximum output level nor be too small to be measured.

[RANGE 2↑()] selects and **sizes** a portion of the full dynamic range for the signal source assigned to an output channel. The portion selected is sized such that the highest possible value for the portion does not exceed maximum output voltage allowed for the given output (+1 mV or +1 V).

ATTN 2↑() further selects and sizes a portion of the **ranged** signal for the + 1 **mV** output to ensure that the signal does not exceed + 1 mV.

Note: ATTN 2↑() functions only for the strip chart recorder output (+1 mV) and acts on the signal AFTER it has been ranged by RANGE 2↑().

For strip chart recorders (analog signal output +1 mV)**:** is attenuated via RANGE 2↑() and ATTN 2↑(). For ATTN 2↑(), each step to a higher value reduces the output signal level (as defined by RANGE 2↑()) by half.

$$\text{+1 mV Output Level} = \frac{\text{Signal}}{2^{\text{RANGE 2↑()}} \quad 2^{\text{ATTN 2↑()}}}$$

For electronic integrators (analog signal output +1 V): is attenuated via RANGE 2↑() from the HP 5890 and must be set at the integrator. Each step to a higher setpoint value decreases the output signal level by a factor of 2 (half the previous level).

$$\text{+1 V Output Level} = \frac{\text{Signal}}{2^{\text{RANGE 2↑()}}}$$

As an example, a key sequence set attenuation and/or range would be:

SIG 1 or SIG 2 RANGE 2↑() *<value>* ENTER

SIG 1 or SIG 2 ATTN 2↑() *<value>* ENTER

The table below gives values permitted for either function, and the output affected.

Valid Setpoints For RANGE 2 and ATTN 2 .

Key	Permitted Setpoints	Affected Output
RANGE 2	0 to13	BOTH **+1 mV& +1 V**
ATTN 2	0 to 10	ONLY **+1 mV**
	OFF ON	

Generally if both an integrator or A/D converter (+ **1 V** output) and chart recorder **(+1 mV** output) are connected to the same signal channel, RANGE 2 should be set properly first for the integrator or computer, then ATTN 2 set appropriately for the chart recorder.

To minimize integration error for an integrator or A/D converter, RANGE 2 should normally be set to the lowest value possible, provided the largest peaks of interest do not exceed 1 volt. Attenuation functions at the integrating device or computer are then used to ensure that plotted peaks remain on scale.

The table below lists maximum detector output producing +1 volt at the **+1V** output for each RANGE 2 setpoint value.

Maximum Detector Signal Producing +1 V Output

RANGE 2	FID & NPD (pA)	TCD (mV, High Gain)	TCD (mV, Low Gain)	ECD (kHz)
0	1.0×10^3	25	800	10
1	2.0×10^3	50	D	20
2	4.0×10^3	D	D	40
3	8.0×10^3	D	D	80
4	1.6×10^4	D	D	160
5	3.2×10^4	D	D	320
6	6.4×10^4	D	D	D
7	1.3×10^5	D	D	D
8	2.6×10^5	D	D	D
9	5.1×10^5	D	D	D
10	1.0×10^6	D	D	D
11	2.0×10^6	D	D	D
12	4.1×10^6	D	D	D
13	8.2×10^6	D	D	D

From the table above, note that for a TCD, RANGE 2 = 0 is suitable for virtually all applications because the entire linear output range of the detector is included. Likewise, RANGE 2 settings from **0** through **5** cover the entire useful output range for an ECD. Only an FID or NPD may require use of the higher RANGE 2 settings.

Turning attenuation off/on

The +1 mV strip chart recorder signal output can be switched off, providing no signal to the data-receiving device. This is often useful in setting the zero position at a connected strip chart recorder.

This is done through the following key sequence:

[SIG 1] or [SIG 2] [ATTN 2 ↑()] [OFF]

After setting the pen to the desired **zero** position at the connected chart recorder, the current attenuation value is restored by pressing [ON].

Entering a new [ATTN 2 ↑()] value overrides `OFF`.

Inverting TCD signal polarity

Note: Expect a baseline shift any time signal polarity is inverted. This may require a baseline reset at a connected integrator or chart recorder.

TCD output is **one** signal representing thermal conductivity difference between two flows (column effluent and reference gas).

- Enter the key sequence:

  [SIG 1] (or [SIG 1]) [A] (or [B])

  to assign the signal to an output channel (either A or B).

- For components giving NEGATIVE-going peaks,

  [DET] [A] (or [B]) [−]

  inverts detector output polarity.

Repeating the entry (or simply pressing [−] again if the TCD is still displayed) inverts polarity again to its original state.

Where a given sample has components giving both positive- and negative-going peaks, a timetable command can be used to invert detector polarity during a run.

For example, to create a timetable event to invert TCD polarity at 1 minute into a run, enter the key sequence:

either detector	[A] or [B]	[DET] [A] [–] [TIME] [1] [ENTER]

Repeating the entry later in the run inverts polarity again to its original state.

Polarity returns to its original state automatically at the end of the run.

Using instrument network (INET)

The "Instrument Network" (INET) is a path for various devices to communicate with each other (data and/or commands). INET permits a group of devices (consisting of a "controller" and some number of data "Producers" and data "Consumers") to function as a single, unified system.

In using the INET function, chromatographic parameters are entered normally through the HP 5890 keyboard. Integration parameters are entered at the controller. Parameters for other devices on the INET loop may be entered at the controller or at their own keyboards. Collectively, the separate sets of parameters constitute a single set of parameters for an "analysis."

In DEFAULT operation, the HP 5890 supplies only "Signal 1" data to the INET loop. That is, HP 5890 data supplied to the INET loop is defined according to the assignment made via [SIG 1]. To use "Signal 2" data instead, signal reassignment is done at the HP 5890.

As an example, a key sequence to assign detector **B** data to the **Signal 1** output channel would be:

[SIG 1] [B] [ENTER]

For more information about INET, refer to the *HP 5890 SERIES* II *Reference Manual* and the reference manuals for your HP integrator/ controller.

Making a Run

Making a Run

This chapter includes information regarding starting and stopping an analytical run, using timetable events, and making a single-column compensation (SCC) run.

Starting/stopping a run

Pressing START starts the oven temperature program, run clock, and timed events.

Also, a remote start relay is momentarily closed to start a remote device such as an integrator. For a strip chart recorder, a tick mark is produced to mark the beginning of a run.

Pressing START lights the green RUN LED. Yellow OVEN LEDs are lit to follow progress through an oven temperature program. The red NOT READY LED lights during a run only if some part of the system becomes **not ready (see** *Status LEDs* on next page).

START aborts a keyboard entry in progress by causing a run to begin immediately. START itself is inactive if the RUN LED is on or blinking.

Pressing STOP terminates a run. For a strip chart recorder, a tick mark is produced to mark the end of a run.

INET start/stop operation

Normally, when the HP 5890 SERIES II (hereafter referred to as HP 5890) and integrator/controller are connected together via INET, START and STOP on either instrument are equivalent. The only exception to this is if INET is turned OFF (LOCAL) at the HP 5890: if INET is off, HP 5890 START and STOP keys are independent of those on the integrator/controller. This permits starting the HP 5890 without simultaneously starting the integrator/controller. (See the *HP 5890 SERIES* II *Reference Manual* for details regarding switching INET on or off).

Note: The HP 5890 STOP key is always active during a run if the run must be aborted at the HP 5890.

Status LEDs

Readiness occurs when the oven is on and at its setpoint temperature, when heated zones that are on are at their respective setpoint temperatures, and when any detector assigned to an output signal channel is **on.**

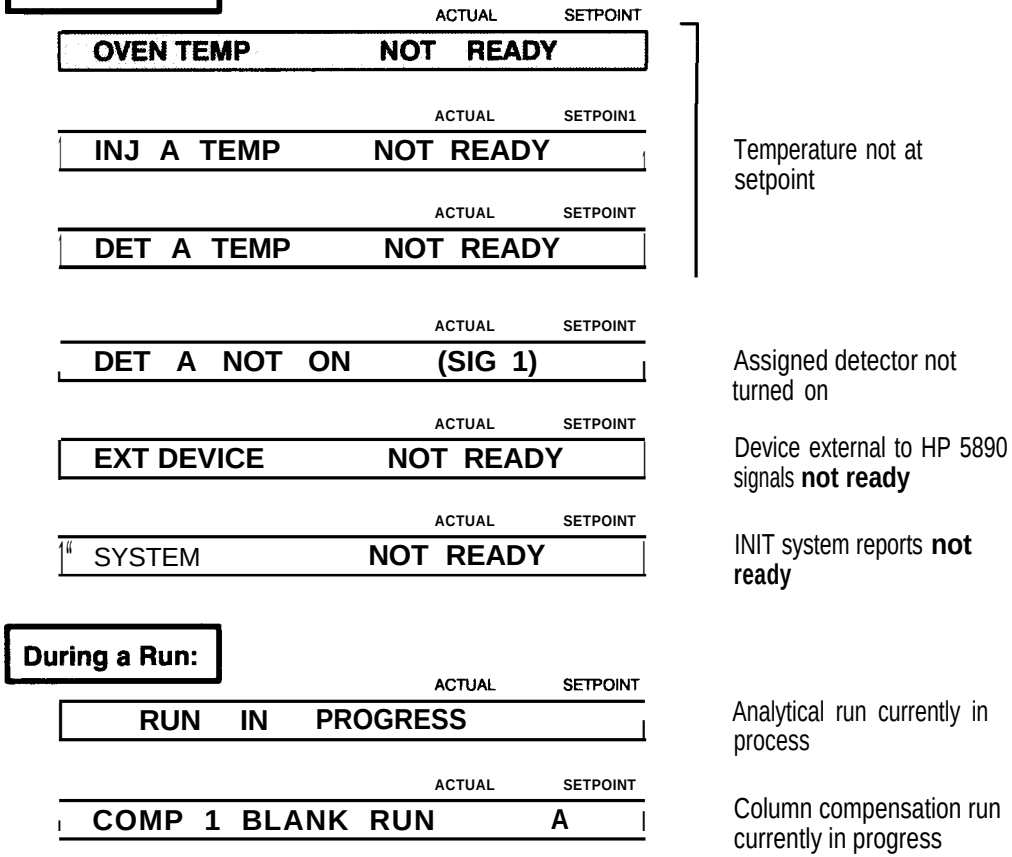
Any temperature not at setpoint causes the red NOT READY LED to be lit until setpoint is achieved.

In addition, there is an external readiness input, and an INET readiness input. If a connected external device is not ready, the NOT READY LED is lit.

If the NOT READY LED is continuously lit, any item(s) preventing readiness can be determined by pressing CLEAR : the display cycles, giving an appropriate message for each item.

CLEAR Displays

Outside a Run:



Temperature not at setpoint

Assigned detector not turned on

Device external to HP 5890 signals **not ready**

INIT system reports **not ready**

During a Run:

Analytical run currently in process

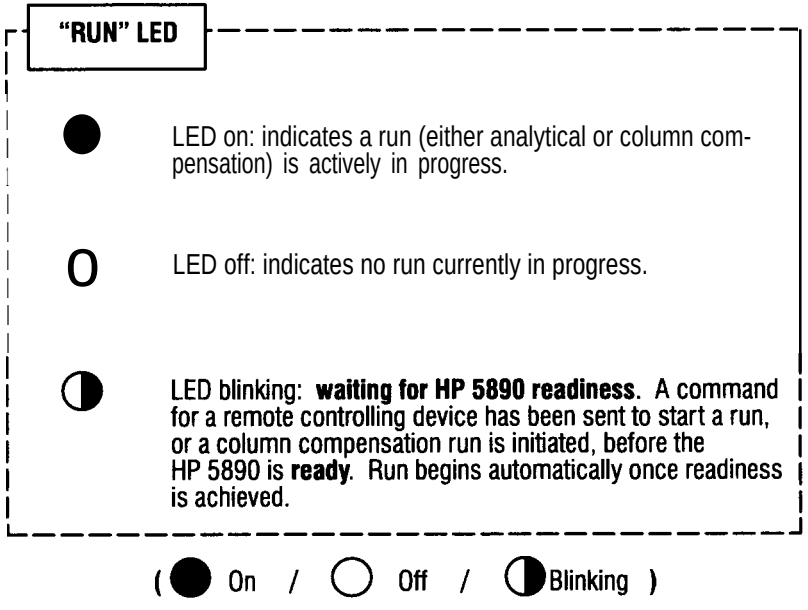
Column compensation run currently in progress

Typical NOT READY **Displays (obtained by pressing** CLEAR **)**

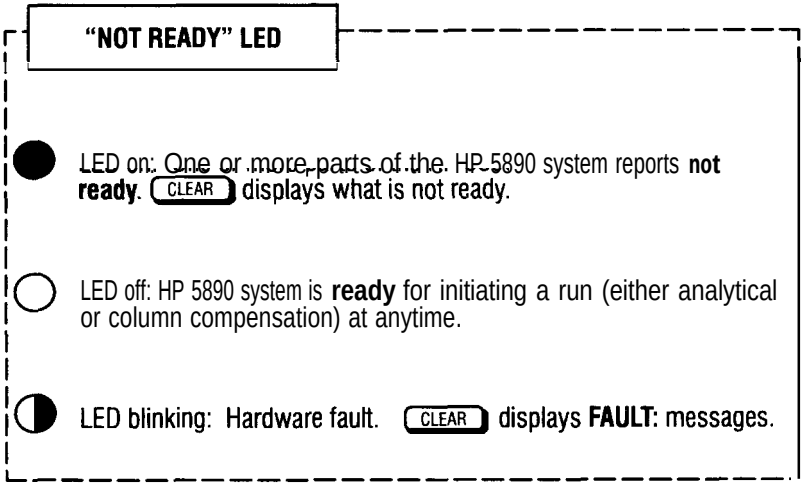
Note that the figure above shows typical normal displays occurring when various parts of a properly operating HP 5890 system are not ready for initiating a run.

HP 5890 READY Display

For the oven and heated zones, their messages cease to be displayed once their respective setpoint temperatures are reached. Once every item is ready, pressing CLEAR results in the display shown above.



Status LED Display



Status LED Display (cont.)

The RUN LED is normally lit continuously whenever a run is in progress (either an analytical or column compensation) and off when not in a run. It flashes when a column compensation run is initiated before the HP 5890 is ready (e.g., oven or zones not at setpoint); the compensation run begins automatically upon readiness.

The RUN LED also flashes on and off in an INET-controlled system in automated operation during times when the HP 5890 is waiting for some device in the system to complete its task before starting the run (e.g., while waiting for automatic sampler operation, report printing, computations, etc). Once the run begins, the LED is continuously lit.

Using the time key

Successively pressing TIME by itself displays various times related to analyses being performed and also accesses a **stopwatch** timer useful in setting gas flow rates, measuring elapsed time between events of interest, etc.

Typical time displays are shown in the figure below. Note that there are three possible functions outside an analytical run (or column compensation run) and three possible during an analytical run (or column compensation run). Each press of TIME rolls to the next function.

Outside a Run:

		ACTUAL	SETPOINT
NEXT	RUN	24.38	MIN
		ACTUAL	SETPOINT
t=5:I0.7		1/t=	0.19
		ACTUAL	SETPOINT
LAST	RUN	15.77	MIN

During a Run:

		ACTUAL	SETPOINT
REMAINING		15.77	MIN
		ACTUAL	SETPOINT
t=	1:50.7	1/t=	0.54
		ACTUAL	SETPOINT
ELAPSED		12.15	MIN

Typical Time Displays

Time displayed for NEXT RUN or LAST RUN does **not** include EQUIB TIME and does not include **cooldown** time after completing an oven temperature program. It is simply total time calculated for the analytical (or column compensation run) itself.

In **stopwatch** mode, both time (to 0.1 second) and reciprocal time (to 0.01 rei[n]-l) are displayed simultaneously. The timer is started by pressing ENTER; it is stopped by pressing ENTER again. Pressing CLEAR **after** the timer is stopped resets the timer.

Note that other instrument functions may be accessed normally (e.g., OVEN TEMP or FLOW) without stopping or resetting the timer simply by pressing the necessary keys. The timer continues to run but is not displayed until TIME is again pressed.

TIME is also used in key sequences to time-program events during a analytical run (see *Using timetable events* in this chapter).

Using single-column compensation

The HP 5890 permits performing a chromatographic **blank run** (run made with no sample injected), storing the data as a baseline profile.

Assuming the baseline profile is consistent from run to run, it maybe subtracted from sample run data to remove baseline drift (usually caused by column bleed).

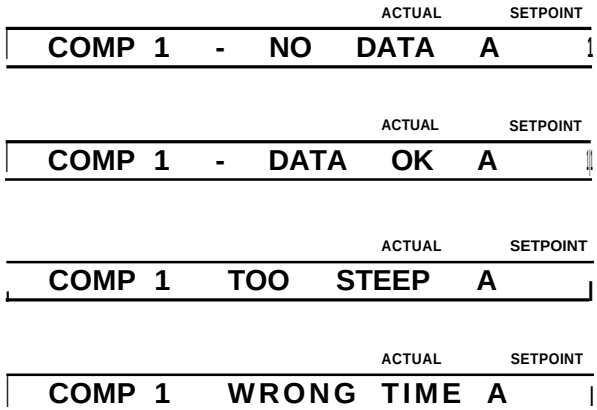
Note: Single-column compensation data is valid only for a specific detector and column combination operating under defined temperature and gas flow rate conditions. Invalid results may occur if conditions by which blank run data is collected are different from conditions used to collect sample run data.

Two separate profiles may be stored (designated by COL COMP1 or COL COMP2), as, for example, one each for two different detectors or two profiles for the same detector but using different chromatographic conditions.

Note: The HP 5890 STOP key is always active during a column compensation run if the run must be aborted at the HP 5890.

Displaying column compensation status

Status of column compensation data is displayed by pressing either COL COMP1 or COL COMP2. The figure below gives typical displays:



No baseline profile data is presently stored for detector **A** in **COMP 1.**

Valid baseline profile data is presently stored for detector **A** in **COMP 1.**

Change in baseline slope exceeds maximum value permitted. Column compensation data may not be valid.

Column compensation run aborted prematurely via STOP. Column compensation data may not be valid.

(Equivalent displays are possible for **COMP 2** and/or detector **B)**

Typical Column Compensation Status Displays

In each display, COMP 1 or COMP 2 echoes the key pressed (COL COMP1 or COL COMP2, respectively); A or B indicates the assigned detector.

Initiating a column compensation run

After entering the oven temperature program to be used for later sample runs, a column compensation run is initiated by first pressing either COL COMP1 or COL COMP2 to display current column compensation status and to designate where the new baseline profile is to be stored.

. If the desired detector (A or B) is displayed, the column compensation run is initiated simply by pressing ENTER.

. If the wrong detector is displayed, press either A or B to assign the desired detector, then press ENTER to initiate the column compensation run.

- . followed by ENTER initiates two parallel column compensation runs using the same oven temperature program and storing a baseline profile for each of the assigned detectors simultaneously.

This option is useful for sample analyses made using different detectors and/or columns but using identical temperature programs.

Note: A device connected via the remote start/HP 5890 ready cable that is started from the HP 5890 by a normal analytical run is **not** started by a column compensation run.

Additional detail concerning functions available at the REMOTE receptacle is found in the *HP 5890 SERIES II Site Prep and Installation Manual.*

Messages listed in the next figure are displayed either while a column compensation run is in progress or if there is a problem preventing the compensation run from starting.

ACTUAL	SETPOINT
COMP 1 BLANK RUN A	

Comp run in progress. In this example, data from detector **A** is stored as **COMP 1** (accessed via COL COMP1).

ACTUAL	SETPOINT
INVALID DURING RUN	

Displayed if an attempt to start a column compensation run is made while a sample run is in progress. No column compensation run is performed.

ACTUAL	SETPOINT
OVEN NOT ON	

The oven is not on. Once the oven is switched on, the column compensation run begins automatically when the oven is equilibrated at its initial temperature setpoint.

ACTUAL	SETPOINT
NO TEMP PROGRAM	

An oven temperature program is not defined: nonzero RATE setpoint value(s) must be entered. The temperature program defined should be that used for sample runs. No column compensation run is performed.

ACTUAL	SETPOINT
DET A NOT ON	

Or detector B, chosen detector not switched on. No column compensation run is performed.

ACTUAL	SETPOINT
DET A NOT INSTALLED	

Or detector B, chosen detector not present. No column compensation run is performed.

ACTUAL	SETPOINT
NO DETECTOR FOUND	

No detector(s) present. No column compensation run is performed.

ACTUAL	SETPOINT
INVALID IN COMP RUN	

Occurs if entering new oven temperature program setpoints is attempted during a column compensation run. Entries are ignored. Also occurs if an attempt is made to start a column compensation run while one is already in progress. The one in progress continues to normal completion.

Typical Column Compensation Message Displays

A column compensation run terminates automatically at completion of its oven temperature program. Any existing baseline profile is erased as data for the new baseline profile is collected and stored.

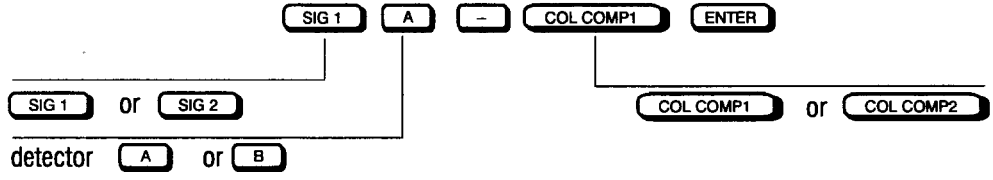
Note that the oven temperature program for a column compensation run follows setpoint values for INIT TIME, RATE, and FINAL TIME as in an analytical run. Data is stored, however, only for RATE and FINAL TIME portions of the temperature program.

A sample run cannot be started (via START) while a column compensation run is in progress. STOP aborts a column compensation run: The baseline profile stored is probably not valid because the oven temperature program will not have reached the FINAL TEMP setpoint. A message WRONG TIME is displayed to indicate a mismatch has occurred between the expected length of time for the run versus the actual time.

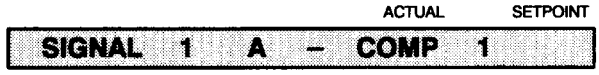
Assigning column compensation data

After baseline data for a given detector (A or B) is stored as either COMP 1 or COMP 2, the column compensation data must be assigned to a specific detector signal. During a run, the compensation data is subtracted from run data for the same detector.

The following key sequence assigns such baseline-corrected data to a particular output channel:



The figure below illustrates the resulting display confirming the assignment:



Column Compensation, Typical Display

Note: There is **no** internal verification by the HP 5890 to ensure that compensation data collected on a given detector is later assigned to be subtracted from the **same** detector via the above key sequence. If subtracting compensation data results in strange baseline behavior:

. Compensation data itself is suspected.

. Data acquired from a different detector has been assigned.

● Chromatographic conditions used for sample analyses are different from those used for the original column compensation run.

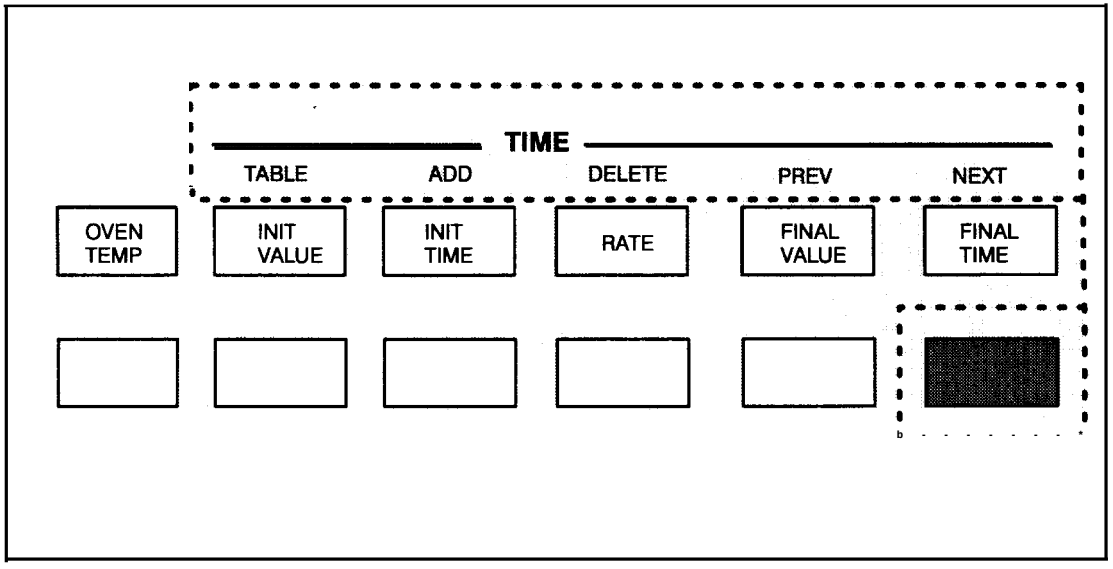
Once assignment is made to a particular output channel, sample analyses are performed in the usual manner; the only difference is that the observed baseline should be relatively free of drift.

Using instrument network (INET)

Information about INET communications is available in Chapter 6, *Controlling Signal Output* and in the reference manuals for your integrator/controller.

Using timetable events

Timetable events are controlled through the keys shown.

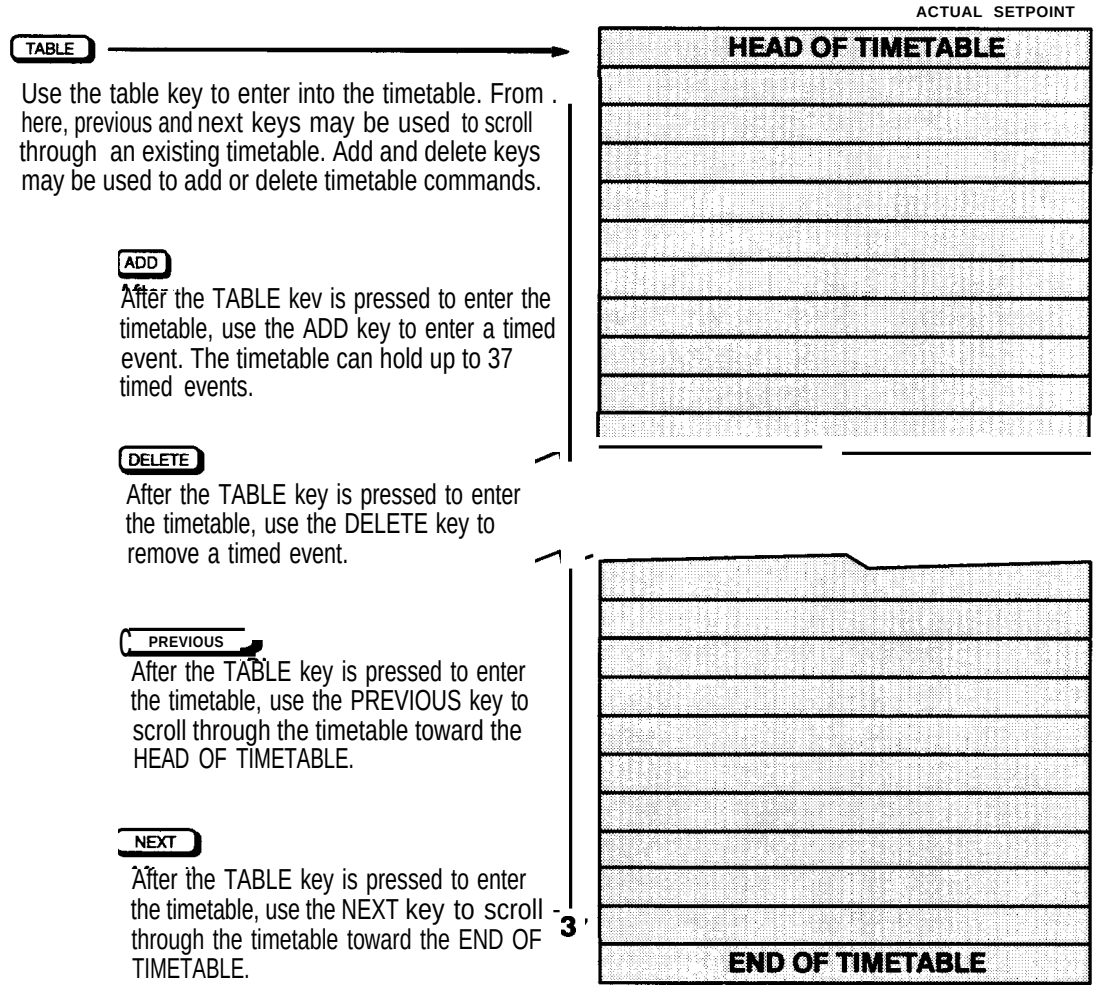


Timetable Control Keys

The following is a list of HP 5890 events that can be controlled during a run.

- **Valves (On/Off)**
- **Signal Switching (On/Off)**
- **Changing TCD Sensitivity (High or Low)**
- **Inverting TCD Polarity (-)** Refer to Chapter 6, *Inverting TCD signal polarity*
- **Split/Splitless Purge Flow On/Off** Refer to Chapter 4, *Setting splitless mode flow*

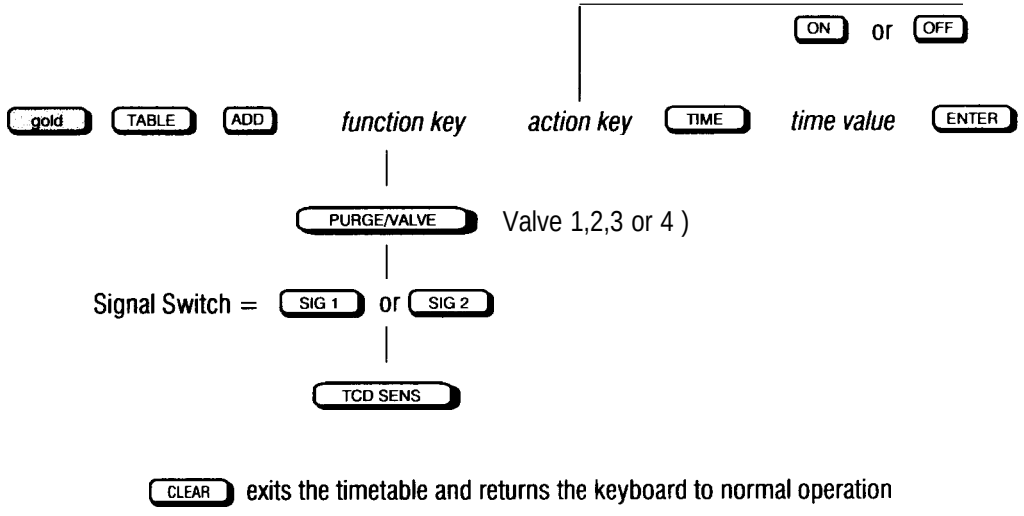
Valves, signal switching and changing TCD sensitivity are covered in this section. Refer to Chapter 6 for information regarding inverting TCD polarity during a run. Refer to Chapter 4 for information regarding turning split/splitless flow on/off during a run.



CLEAR **After timetable entries have been made, (or** whenever working inside the timetable) use the CLEAR key to exit timetable programming.

Note: The clear key does not delete any timetable events.

An example key sequence to create a timetable event:



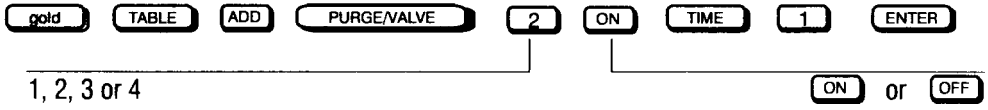
Note: Refer to "Inverting TCD Signal Polarity" in Chapter 6 for time programming TCD polarity.

Turning valves on/off during a run

For information on controlling splitless purge flow during a run, refer to Chapter 4, *Setting splitless mode flow*.

Control of up to four gas/liquid sampling valves (designated as valve 1,2,3, and 4) may occur in either of two ways. The operator may switch the valves manually whenever it is desirable via keyboard entry, or more conveniently, the valves can be switched **on** and **off** during a run via the HP 5890's timed events table.

For example, to create a timetable event to turn valve 2 on at 1 minute into a run, enter the key sequence:



Note: If the valve is already in the position where a command instructs it to switch, no action will occur.

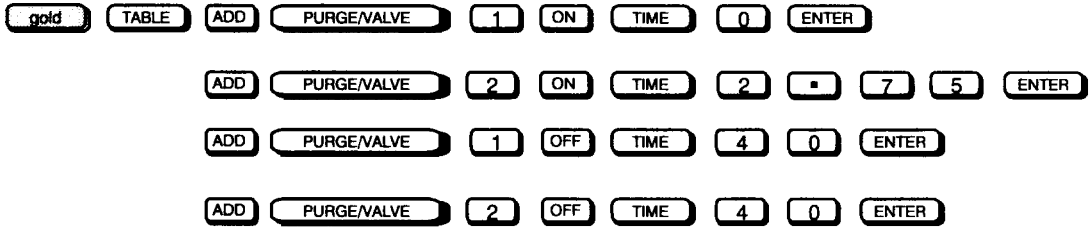
The designated channels (1, 2, 3, or 4) are determined solely by the wiring connections to the valve box.

A valve will reset automatically at the end of each run if a valve timetable is set. If the last switching mispositioned the valve and the valve does not reset, reset the valve position manually before starting a new run.

One way of resetting the valve automatically after the useful run time is to program the valve.

For example, on a two-valve system, where valve 1 is a gas sampling valve and valve 2 is used for venting, it may be desirable to: 1) inject from valve 1 at the beginning of the run (run time 0.00); 2) vent the last part of the sample, using valve 2, at 2-3/4 minutes (run time 2.75); or 3) relax both valves just prior to the end of the run (determined to be a run time of 40.00).

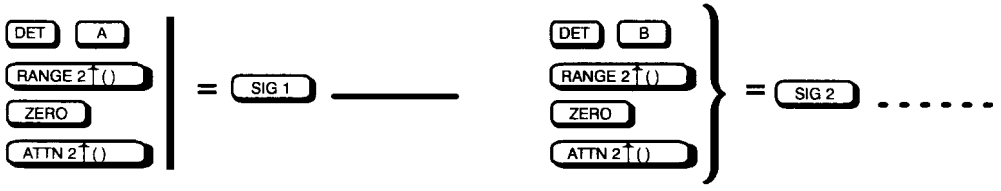
To perform the above example, enter the following commands:

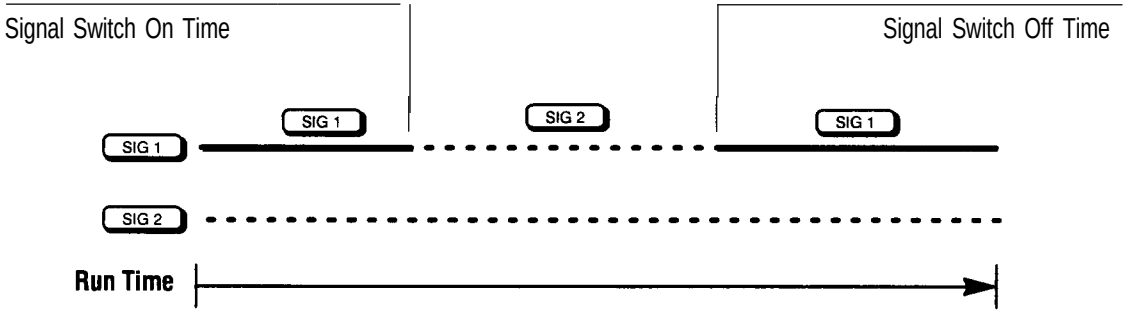


Switching signals during a run

Some analyses require the use of more than one detector to completely characterize a given sample. In situations were the analytical system can be configured to avoid coelution, switch signals during a run to integrate the output into a single channel system. Signal switching can be accessed only as a timetable event.

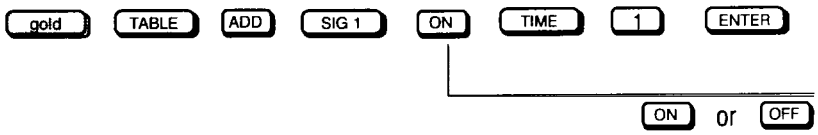
Switching signals replaces the SIG 1 definition to whatever is currently assigned to SIG 2 for a specified period of time.





Note: The ATTN 2↑() key on the HP 5890 has no effect when using an electronic integrator. Attenuation is controlled locally at the integrator.

For example, to create a timetable event to switch signals at 1 minute into a run, enter the key sequence:



During a run, at the signal switch on time, SIG 1 will switch automatically to monitoring the device assigned to SIG 2. The run will continue in this manner until a signal switch off time is reached or the run ends. Signal assignments reset automatically at the end of each run.

Depending on the analyses, baseline upsets maybe seen when signals are switched. These upsets will become more of a problem when high sensitivity is required. Adjust ATTN 2 [ ], ZERO, or RANGE 2 [ ] if necessary to minimize the upset.

Changing TCD sensitivity during a run

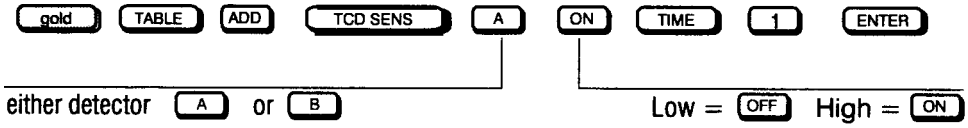
Two TCD sensitivity (signal amplification) settings are available, controlled through the keyboard (ON =HI and OFF =LOW).

The high-sensitivity setting increases sensitivity (area counts observed) by a factor of 32 and is usable in applications where component concentrations are < 10%.

Components that are more concentrated may exceed the output range for the TCD, causing flat-topped peaks. If this occurs, the low sensitivity setting should be used instead.

The sensitivity setting may be changed from one setting to the other at any time during a run through a timetable event.

For example, to create a timetable event to change the TCD sensitivity from Low to Hi at 1 minute into a run:



Sensitivity returns to its original state automatically at the end of the run.

Modifying timetable events

FUNCTION	TIME
VALVE 1 ON	1.00

Example Timetable Event

Timetable events can be changed in three ways: by adding new entries, by deleting existing entries or by modifying the time value of a timed event.

New entries are added by using the same key sequences used to create a table in the first place:

gold TABLE ADD folowed by a valid timetable event

To **delete** a single timetable event, first display the event by pressing:

gold TABLE (followed by PREVIOUS or NEXT when needed)

While the particular event is displayed, delete the event from the timetable by pressing:

DELETE ENTER The instrument will respond, DELETED.

To **modify the time** associated with an event, first display the event by pressing:

gold TABLE (followed by PREVIOUS or NEXT when needed)

To modify the time press:

TIME followed by the new time value, ENTER The instrument will respond, MODIFIED.

For example, to change VALVE 1 ON 1.00 to VALVE 1 ON 2.00, (while VALVE 1 ON 1.00 is displayed) the key sequence is:

TIME 2 ENTER

The result is valve 1 will turn on at 2 minutes rather than 1 minute.

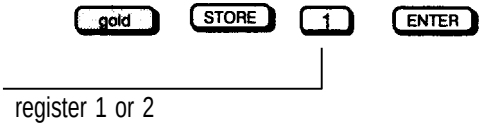
Storing and Loading HP 5890 Series 11 Setpoints

Storing and Loading HP 5890 Series II Setpoints

Up to two sets of GC setpoints may be stored in the HP 5890 Series II (hereafter referred to as HP 5890). GC setpoints include any entry made through the HP 5890 keyboard. These sets of GC setpoints are stored in storage registers designated as 1 or 2.

Storing GC setpoints

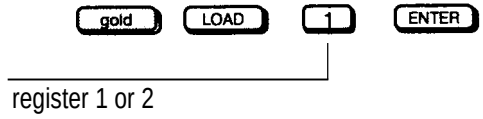
To store a set of setpoints currently in the HP 5890 into a storage register, use the following key sequence:



Loading GC setpoints

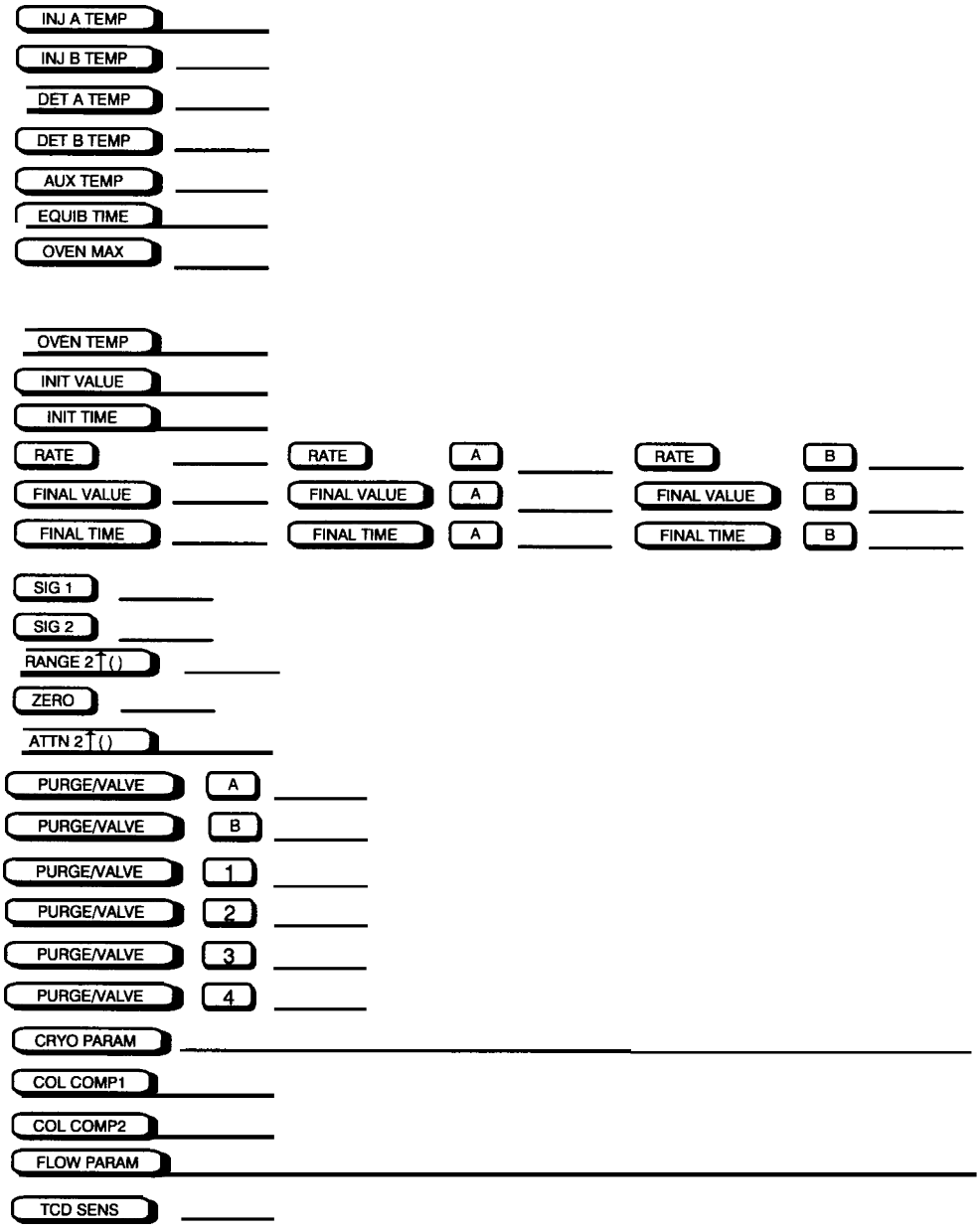
WARNING Be careful when loading GC setpoints from a storage register. Stored setpoints may turn detectors on or set high oven temperatures which, without proper gas flow, could damage a detector or column.

To load a set of setpoints already stored in one of the storage registers, the key sequence is:

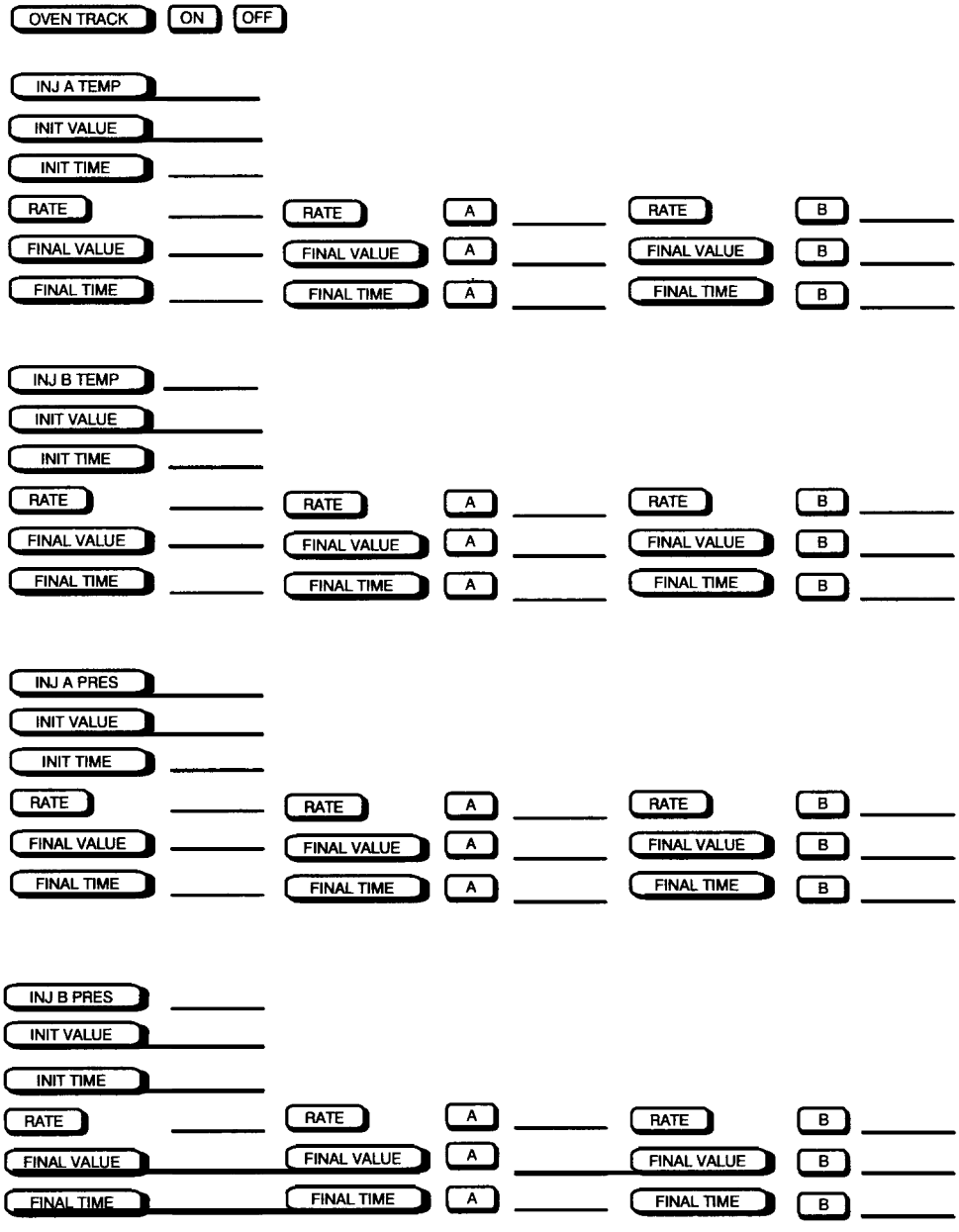


Loading setpoints replaces all the setpoints currently defined in the HP 5890 with the setpoints in the storage register selected.

As a guide to recording GC setpoints stored in storage registers, the following pages may be photocopied and used to record a list of setpoints before they are stored. Keep these lists for your records when preparing to load setpoints.



Storage Setpoint Log



Storage Setpoint Log (continued)

Time Table Events:

Storage Setpoint Log (continued)

Controlling Valves

Controlling Valves

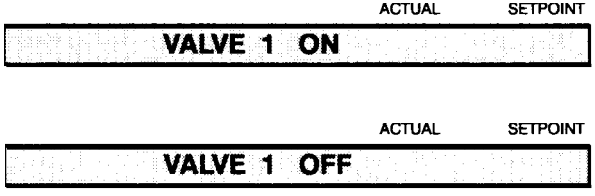
Control of up to four valves (designated as valve 1,2,3, and 4) may be accomplished in two ways.

. During a run (see Chapter 7, *Turning valves on/off during a run)*

. Manually through keyboard entry as described in this chapter

Note: If the valve is already in the position where a command instructs it to switch, no action will occur.

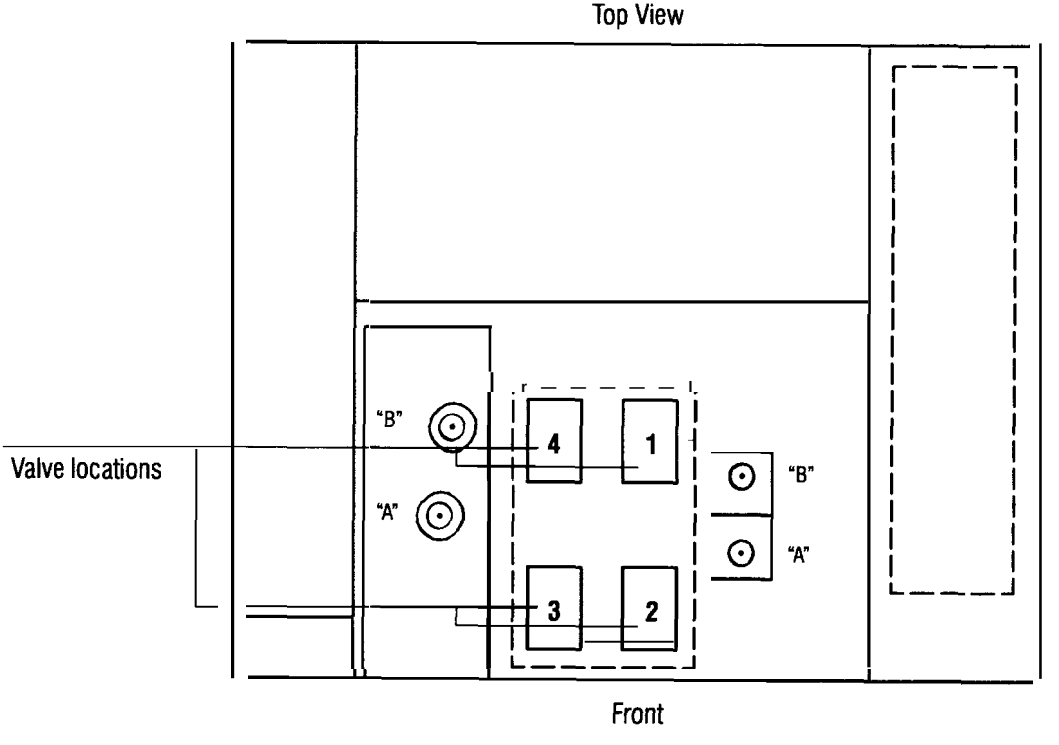
The following figure illustrates some examples of the HP 5890 SERIES II (hereafter referred to as HP 5890) alphanumeric display for listing current valve status or verifying the timed events table to switch the valve during a run.



Typical Valve Status Displays

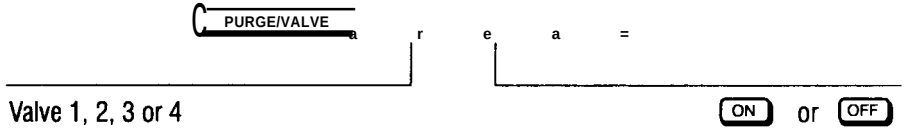
Turning valves on/off manually

The designated channels (1, 2, 3, or 4) are determined solely by the wiring connections to the valve box. However, often valves will be located as shown in the figure below.

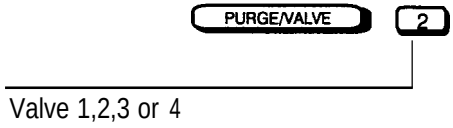


Valve Locations

Valves may be switched from the keyboard at any time by pressing the key sequence:



To display the current status of a valve, press:



If the HP 5890 display is already displaying the appropriate addressed valve, the operator need only press the ON or OFF key to activate or relax the displayed valve.

A valve resets automatically at the end of each run. If the last switching mispositioned the valve for the start of the next run, the valve position will reset at the end of the run.

Using Electronic Pressure Control

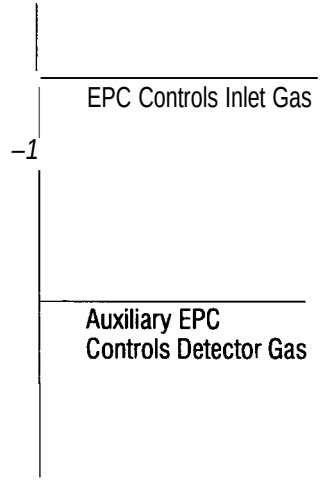
Using Electronic Pressure Control

What is electronic pressure control?

The electronic pressure control (EPC) option for the HP 5890 Series II Plus GC allows you to control the inlet and auxiliary gases from the keyboard.

You access the inlet and auxiliary detector gases by pressing one of the key sequences shown in the following table. The keys you press depend on how your GC is configured. For example, if auxiliary channel C is programmed to control the makeup gas for detector A, you would access the auxiliary EPC channel C to access control of that gas.

Press:	To Access:
gold + INJ A TEMP	*Injector A pressure*
gold + INJ B TEMP	*Injector B pressure*
gold + A	*Auxiliary EPC channel C*
gold + B	*Auxiliary EPC channel D*
gold + COL COMP1	*Auxiliary EPC channel E*
gold + COL COMP2	*Auxiliary EPC channel F*



With EPC, inlet and detector pressures can be either constant or programmed. The following sections describe the benefits of using EPC for inlets and detectors.

Using electronic pressure control with inlets (EPC)

EPC of inlets provides very accurate and precise control of column head pressure, typically resulting in retention time reproducibility of better than 0.02% RSD when no column effects are present. With EPC, you can set constant pressure and pressure programs through the keyboard. Inlet pressures can also be set to maintain a desired column flow rate when the column parameters have been entered.

Using electronic pressure control with detectors (auxiliary EPC)

Auxiliary EPC allows you to control detector gases electronically. With auxiliary EPC, you can set constant pressure and pressure programs through the keyboard. Auxiliary EPC is provided by the combination of new, electronically controlled flow modules for gases and the PC board capability to control those modules.

Auxiliary EPC of detector gases allows you to program the makeup gas to optimize a detector's performance. With auxiliary EPC, you can control:

- All detector gases, including makeup, carrier, and fuel gases
- Gas flow to an external sampling device, such as a purge and trap or headspace system
- Gas flow through the split vent of a split/splitless inlet, which can save gas and optimize the operation of the inlet (see *Operating the Gas Saver for the Split/Splitless Inlet* in this chapter)

The following table shows the multiple uses of EPC for inlets and auxiliary EPC for detectors:

Uses for EPC and Auxiliary EPC

	EPC	Auxillarv EPC
Head Space	●	●
Purge and Trap	●	●
Gas Saver		●
Thermal Resorption	●	●

Note: All HP 5890 instruments built before July 1, 1990, will display the message *Change EPC ROM* when used with EPC. When this occurs, contact a Hewlett-Packard service representative to upgrade and install the new ROM.

For additional EPC information, see the HP application note Analysis of *Oxygenates in Gasoline, Including ETBE and TAME, Using Dual-Channel Electronic Pressure Control,* HP Application Note 228-174, publication no. (43) 5091-4701E.

This chapter is divided into several sections, including general EPC instructions, optimization information for inlets, and optimization information for detectors. Specifically, this chapter provides operating and optimization information for the following EPC systems:

. Split/splitless capillary inlet with EPC

. Septum purged packed Inlet with EPC

. Auxiliary EPC of detector gases

● Auxiliary EPC with Gas Saver for the split/splitless inlet

. Auxiliary EPC with external sampling devices

Operating information for the programmable cool on-column inlet is provided in a separate manual included with the manual set. The following table describes the features that are available for specific uses and applications of EPC:

Features of Electronic Pressure Control

Pressure Controlled Function	Constant Pressure	Pressure Programs	Constant Flow Mode	Set Mass Flow Rate	Flow** Programs	Set Avg. Linear Velocity	Vacuum Comp.*
Split /Splitless Cap. Inlet (Carrier Gas)	●	0	●	●	●	●	●
Septum PP Inlet (Carrier Gas)	●	0	●	●	●	●	●
Programmable Cool On-Column Inlet	●	0	●	●	●	●	●
Auxiliary EPC (Detector Gas)	●	0					
Auxiliary EPC (Gas Saver)	●	0					
Auxiliary EPC (General Purpose)	●	0					

*Auxiliary inlet channel will calculate the vacuum compensation.
**You enter the required pressures.

Note: Constant flow mode is recommended for use with 530µ capillary columns. For packed columns, you must calibrate for each individual column to correct for different column lengths and possible settling of the column packing.

Safety shutdown for electronic pressure control

Systems equipped with EPC have a safety shutdown feature to prevent gas leaks from creating a safety hazard. The safety shutdown feature is designed to prevent an explosive concentration of hydrogen carrier gas from accumulating in the GC oven if a column breaks.

Back pressure regulated inlet systems (split/splitless capillary inlet) with EPC cannot detect a column leak, however, because a column leak would occur before the gas reaches the EPC valve. These systems limit the leak rate into the oven using the total flow controller. Under these conditions, hydrogen diffusion out of the oven is fast enough to keep hydrogen concentration below the 4.l% lower explosion limit.

What happens during **electronic pressure control safety shutdown?**

If the system cannot reach a pressure setpoint, the system beeps. After about 2 minutes, the beeping stops and the following message appears on the display:

The system shuts down by entering a pressure setpoint of zero for the affected channel, turning off all heated zones, and locking the keyboard. The table on the following page summarizes the safety shutdown.

Summary table of safety shutdown

What channels can shut down?	The system can shut down all inlet and auxiliary channels.
When does the system start beeping?	The beeps start 10 seconds after the pressure falls below 0.1 psi of setpoint.
What is the frequency of the beeping?	The system beeps at 10,40,60,70,79,87,94, 100,105, 109, 112,114, 115,116,117, 118,119, and 120 seconds.
When does the system stop beeping?	The beeps stop 120 seconds after the system falls short of the setpoint pressure. The actual safety shutdown begins.
What happens after the safety shutdown occurs?	1. The keyboard locks. 2. All heated zones are turned off. 3. The oven fan is turned off. 4. The GC display shows the shutdown message. 5. The other pressure setpoints do not change. 6. The setpoint of the affected EPC channel is set to 0.0.

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

Setting inlet pressure using electronic pressure control

If your GC is equipped with EPC, you can set constant pressure or create a pressure program with multiple ramps. The following procedures will show you how to:

. Zero the pressure sensor in all channels and repressurize the system

. Set and maintain a constant pressure

● Set a pressure program using one or two ramps

● Check your pressure program

For more information on setting inlet pressures, see Chapter 4, *Setting Inlet Pressure.*

Zeroing the pressure

The EPC system is zeroed before shipping, but you should check it periodically especially when ambient laboratory conditions change dramatically Zero the instrument 30 to 60 minutes after the system has heated up to allow for electronic drift.

To zero an EPC channel:

1. Turn off the inlet and detector gases. With zero pressure, remove the inlet septa and repressurize the inlet.

   Note: If the detector gas valves are closed, you will not be able to repressurize the system.

   Note: When EPC is part of a GC-MS system, zero the pressure when either the MS pump is off or the column is not connected to the inlet. Otherwise, the vacuum pump will lead to miscalibration.

2. Use the steps below to zero channels A through F:

- To zero channel A:

  a. Press: gold INJ A TEMP 0 . 0 ENTER
  Sets the inlet A pressure to 0.0

  Allow enough time for the column to completely depressurize.

  b. Press: gold 2 ENTER *value* ENTER

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

- To zero channel B:

  a. Press: gold INJ B TEMP 0 . 0 ENTER
  Sets the inlet B pressure to 0.0

  Allow enough time for the column to depressurize completely.

  b. Press: gold 3 ENTER *value* ENTER

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

- To zero channel C:

  a. Press: gold A 0 . 0 ENTER
  Sets channel C pressure to 0.0

  Allow enough time for the system to repressurize completely.

  b. Press: gold 4 ENTER *value* ENTER

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

- To zero channel D:

  a. Press: gold B 0 . 0 ENTER
  Sets channel D pressure to 0.0

  Allow enough time for the system to repressurize completely.

  b. Press: gold 5 ENTER *value* ENTER

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

. To zero channel E:

  a. Press: gold COL COMP1 0 . 0 ENTER
  Sets *channel* E *pressure to 0.0*

  Allow enough time for the system to repressurize completely.

  b. Press: gold 6 ENTER *value* ENTER

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

. To zero channel F:

  a. Press: gold COL COMP2 0 . 0 ENTER
  Sets channel F pressure to 0.0

  Allow enough time for the system to repressurize completely.

  b. Press: gold 7 ENTER *value* ENTER

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

Setting constant flow mode

While using constant flow mode, the pressure will change if the oven temperature changes to keep the flow constant. When you select constant flow, you can set an initial pressure (at oven initial temperature) and the GC maintains the initial flow throughout the run by adjusting pressure continuously and automatically. This example shows how to set the inlet B pressure at 10 psi.

1. Use the following steps to turn on the constant flow mode:

   a. Press: gold FLOW to access the flow parameters display.

   b. Continue to press FLOW until you see the constant flow display.

	ACTUAL	SETPOINT
EPP B CONST FLOW OFF		

   c. Press: ON to turn the constant flow mode on.

2. Press: gold INJ B TEMP 1 0 ENTER

   Sets the inlet B pressure to 10 psi

	ACTUAL	SETPOINT
EPP B	10.0	10.0

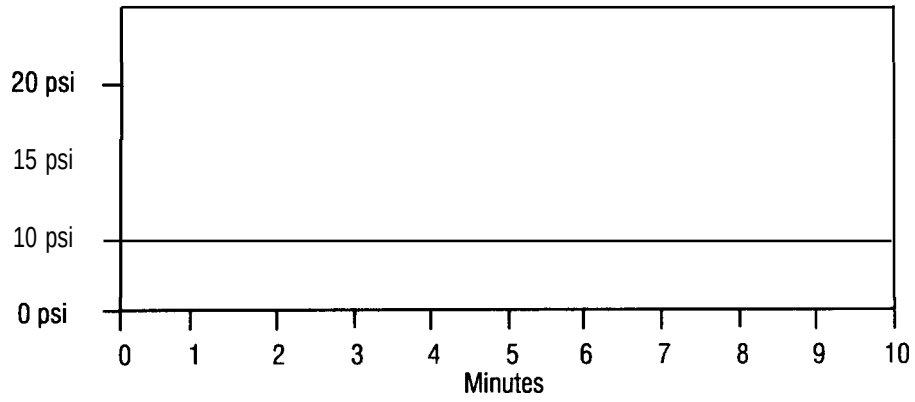
The GC display looks like this

3. Press: INIT TIME 6 5 0 ENTER

   Sets the initial time to 650 minutes (max.)

	ACTUAL	SETPOINT
B: INIT TIME	**650.00**	

The GC display looks like this



Inlet B at Constant Pressure

Note: Inlet B will stay at 10 psi until the oven temperature changes. Then the pressure will increase to keep the flow constant.

Setting inlet pressure programs

The run time of the analysis is determined by the oven temperature program. If the inlet pressure program is shorter than the oven temperature program, the inlet pressure does not remain at the last value but goes into constant flow mode for the remainder of the run. If the oven temperature is changing, then the pressure will also change. To prevent a pressure program from going into constant flow mode, set the pressure program longer than the oven temperature program.

The following procedure shows how to create a pressure program with three pressure ramps for inlet B.

1. Turn the constant flow mode off.

   Note: For more information on setting constant flow mode, see *Using constant mass flow mode for inlets* later in this chapter.

2. Use the following steps to program the first pressure ramp:

The first pressure ramp starts at 10 psi for 1 minute, then ramps at 5 psi/rein to 20 psi and remains there for 2 minutes.

The first pressure ramp starts at 10 psi for 1 minute, then ramps at 5 psi/rein to 20 psi and remains there for 2 minutes.

a. Press: gold INJ B PRES INIT VALUE 1 0 ENTER
Sets inlet B pressure to 10 psi

b. Press: INIT TIME 1 ENTER
Sets the initial time at 1 minute

c. Press: RATE 5 ENTER
Sets the ramp rate at 5 psi/min

d. Press: FINAL VALUE 2 0 ENTER
Sets the final pressure at 20 psi

e. Press: FINAL TIME 2 ENTER
Sets the final time at 2 minutes

3. Use the following steps to program the second pressure ramp:

The second pressure ramp starts at 20 psi and ramps at 2 psi/rein to 26 psi. It remains at 26 psi for 2 minutes.

a. Press: RATE A 2 ENTER
Sets the second ramp rate at 2 psi/min

b. Press: FINAL VALUE A 2 6 ENTER
Sets the second final pressure at 26 psi

c. Press: FINAL TIME A 2 ENTER
Sets the second final time at 2 minutes

4. Use the following steps to program the third pressure ramp:

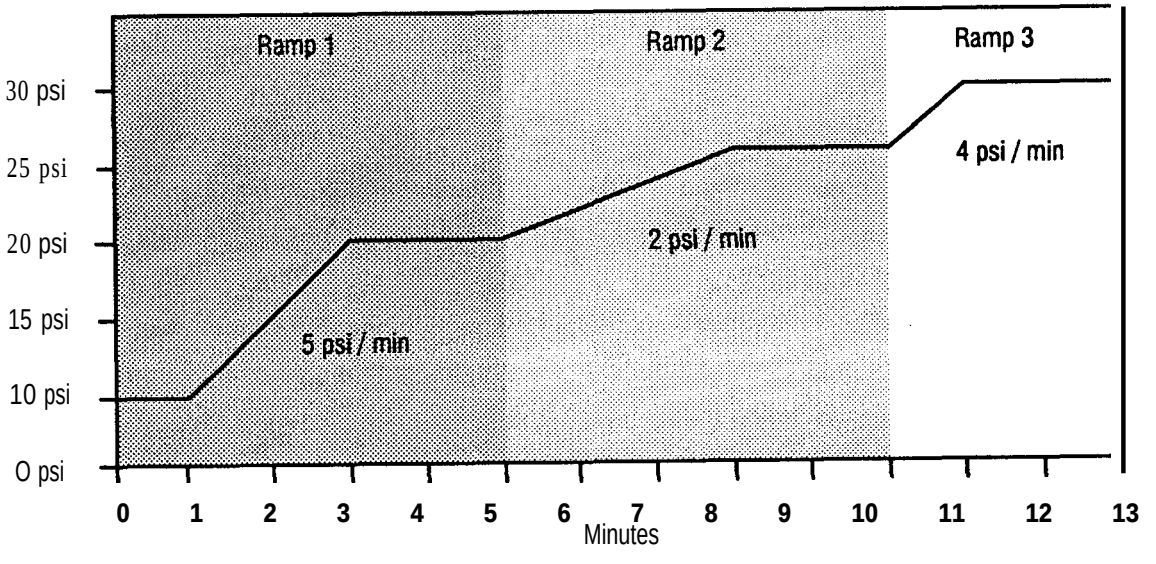
   The third pressure ramp starts at 26 psi and ramps at 4 psi/min to 30 psi. It remains at 30 psi for 2 minutes.

   a. Press: RATE B 4 ENTER
   Sets the third ramp rate at 4 psi/min

   b. Press: FINAL VALUE B 3 0 ENTER
   Sets the third final pressure at 30 psi

   c. Press: FINAL TIME B 2 ENTER
   Sets the third final time at 2 minutes

   The following graph shows the entire three-ramp pressure program.

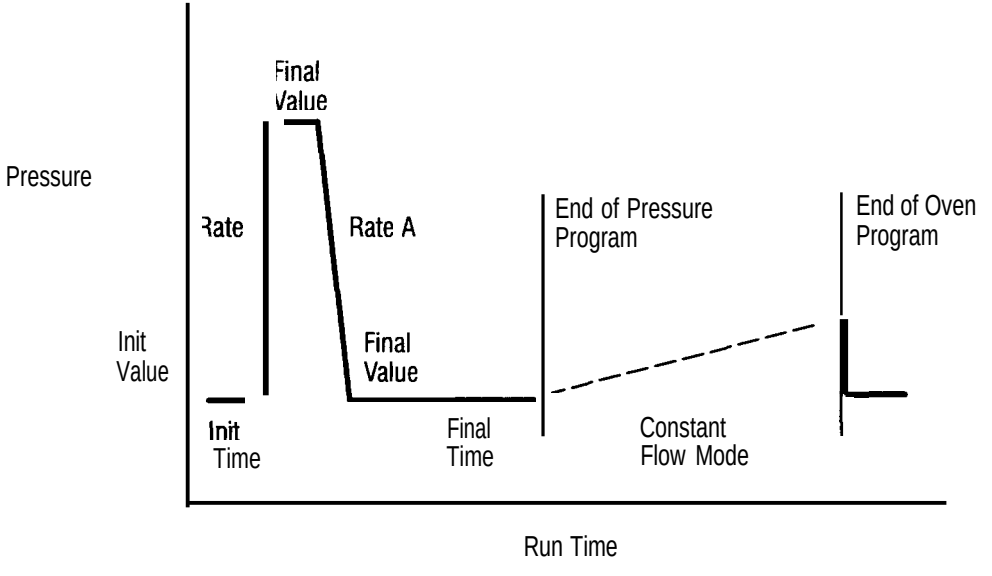


Inlet B with 3 Pressure Ramps

Checking inlet pressure programs

1. Display the pressure program by pressing any pressure program key followed by ENTER.
2. Press ENTER successively to scroll through and view the pressure 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 goes into constant flow mode for the remainder of the run. To prevent this, make sure that the inlet pressure program is equal to or longer than the oven program.



Pressure Program

Setting pressure using auxiliary electronic pressure control

Auxiliary EPC is generally used for applications other than the control of carrier gas to the column. The auxiliary channels (labeled C through F) do not use the pressure versus flow calculations that the inlet channels have. Only pressure setpoints and programs are entered, with flow values determined from the calibration curves established.

For applications such as detector gas control, where the restriction is provided mainly by a flow restrictor in the detector block, the calibration curves are described by the following equation:

$$F = kx\ P^{M}$$

Note: This is the equation used by the HP 3365 ChemStation for pressure versus flow calculations with the auxiliary EPC channels. Values for the constants k and M are displayed on the auxiliary pressure programs screen of the ChemStation when the calculations are carried out. For term definitions, see your ChemStation manual.

How do I access auxiliary electronic pressure control?

To access the auxiliary EPC channels, use the following keys at the GC keyboard:

Press:	To Access:
gold + A	*Auxiliary EPC channel C*
gold + B	*Auxiliary EPC channel D*
gold + COL COMP1	*Auxiliary EPC channel E*
gold + COL COMP2	*Auxiliary EPC channel F*

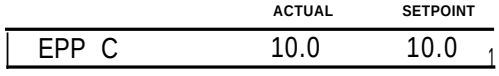
Caution The pressure that you program at the keyboard is the pressure the HP 3365 ChemStation uses in its calculations. When operating under low pressure, such as 15 psi, be sure to program the same pressure (15 psi) at the keyboard. If the rate entered at the keyboard is higher, the ChemStation will base its calculations on that rate, which may not be the accurate flow through your system.

Setting constant detector pressure

This example shows how to set the auxiliary EPC channel C pressure at 10 psi. For additional operating information, see Chapter 5, *Operating Detector Systems.*

1. **Press:** gold A 1 0 ENTER

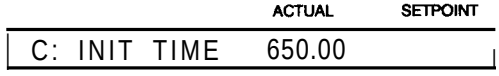
   Sets auxiliary EPC channel C pressure to I0 psi



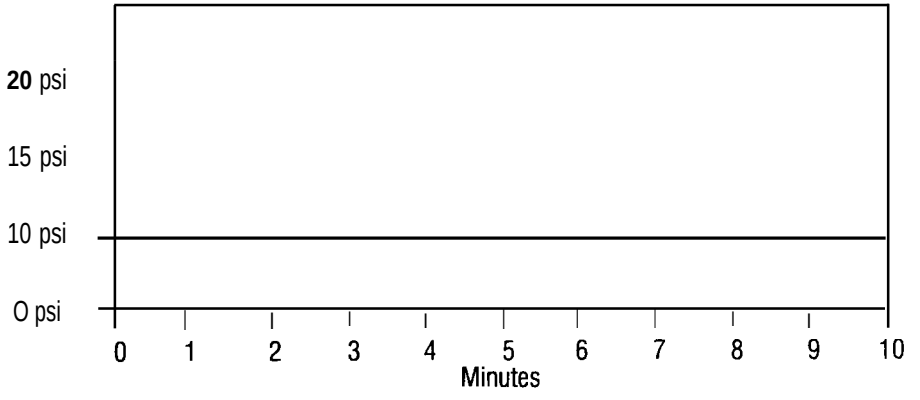
   The GC display looks like this

2. **Press:** INIT TIME 6 5 0 ENTER

   Sets the initial time to 650 minutes (max.)



   The GC display looks like this



Auxiliary EPC Channel C at Constant Pressure

Setting detector pressure programs

The following procedure shows how to create a pressure program for auxiliary EPC channel C with three pressure ramps. For additional operating information, see Chapter 5, *Operating Detector Systems*.

1. Use the following steps to program the first pressure ramp:

   The first pressure ramp starts at 10 psi for 1 minute, then ramps at 5 psi/min to 20 psi and remains there for 2 minutes.

   a. Press: gold A INIT VALUE 1 0 ENTER
   Sets auxiliary EPC channel C initial pressure to 10 psi

   b. Press: INIT TIME 1 ENTER
   Sets the initial time at 1 minute

   c. Press: RATE 5 ENTER
   Sets the ramp rate at 5 psi/min

   d. Press: FINAL VALUE 2 0 ENTER
   Sets the final pressure at 20 psi

   e. Press: FINAL TIME 2 ENTER
   Sets the final time at 2 minutes

2. Use the following steps to program the second pressure ramp:

The second pressure ramp starts at 20 psi and ramps at 2 psi/min to 26 psi. It remains at 26 psi for 2 minutes.

a. Press: RATE A 2 ENTER
Sets the second ramp rate at 2 psi/min

b. Press: FINAL VALUE A 2 6 ENTER
Sets the second final pressure at 26 psi

c. Press: FINAL TIME A 2 ENTER
Sets the second final time at 2 minutes

3. Use the following steps to program the third pressure ramp:

The third pressure ramp starts at 26 psi and ramps at 4 psi/min to 30 psi. It remains at 30 psi for 2 minutes.

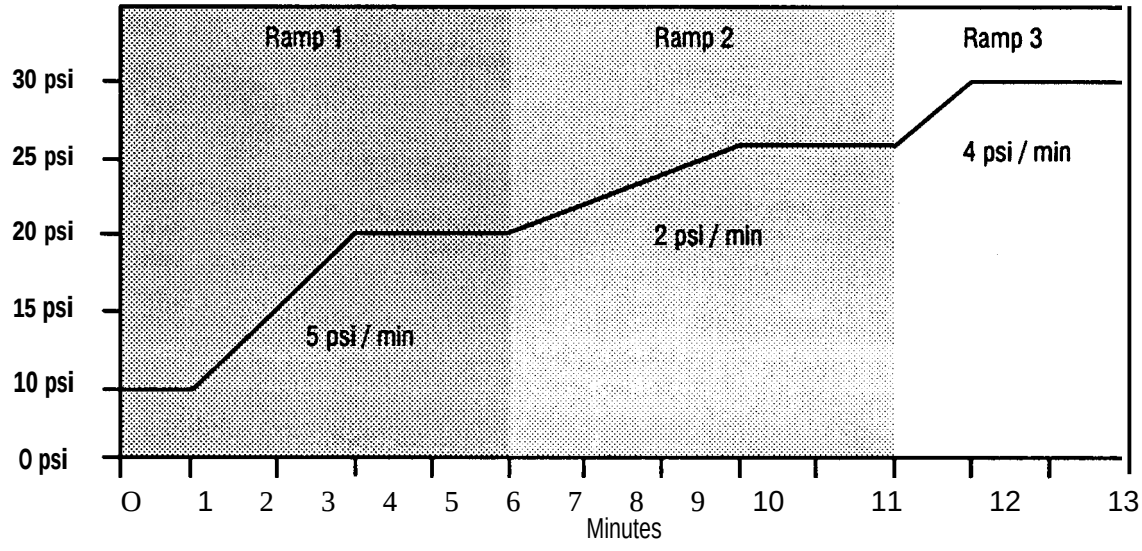
a. Press: RATE B 4 ENTER
Sets the third ramp rate at 4 psi/min

b. Press: FINAL VALUE B 3 0 ENTER
Sets the third final pressure at 30 psi

c. Press: FINAL TIME B 2 ENTER
Sets the third final time at 2 minutes

The following graph shows the entire three-ramp pressure program.

Auxiliary EPC Channel C with Three Pressure Ramps



Checking Detector Pressure Programs

To check your pressure program, press ENTER successively to scroll through and view the program immediately after setting it.

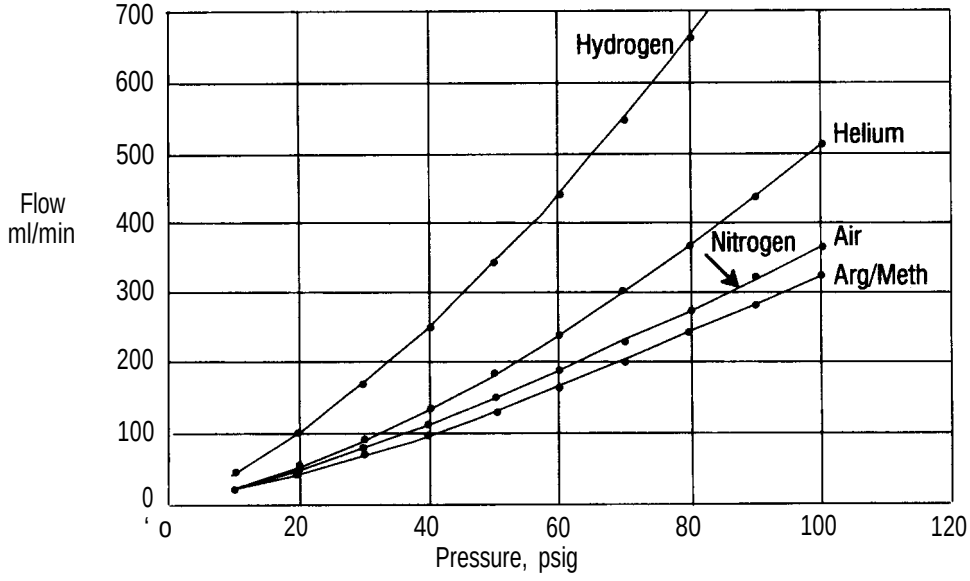
Suggested ranges for operating auxiliary electronic pressure control

The following graphs show the ranges that Hewlett-Packard suggests to optimize the operation of auxiliary EPC for detectors. The graphs show the flow restrictor you will need (the restrictors are identified by colored dots) and the corresponding pressure versus flow relationship. Use the table that most closely corresponds to the gas type you will use in your analysis.

Auxiliary EPC Restrictor Kit

19234-60600 Green and Brown Dot
Computed nominal values at ambient temperature of 21°C and pressure of 14.56 psia

Pressure (kPa)	Pressure (psig)	Helium Flow (ml/min)	Nitrogen Flow (ml/min)	Hydrogen Flow (ml/min)	Air Flow (ml/min)	Argon/Meth Flow (ml/min)
69.0	10	**22**	20	42	21	19
137.9	20	50	45	99	45	40
206.8	30	87	76	170	76	65
275.8	40	131	111	251	110	95
344.7	50	182	150	344	148	128
413.7	60	239	190	442	188	164
482.6	70	300	232	549	229	202
551.6	80	366	275	666	273	242
620.5	90	437	321	786	318	282
689.5	100	513		901	363	324

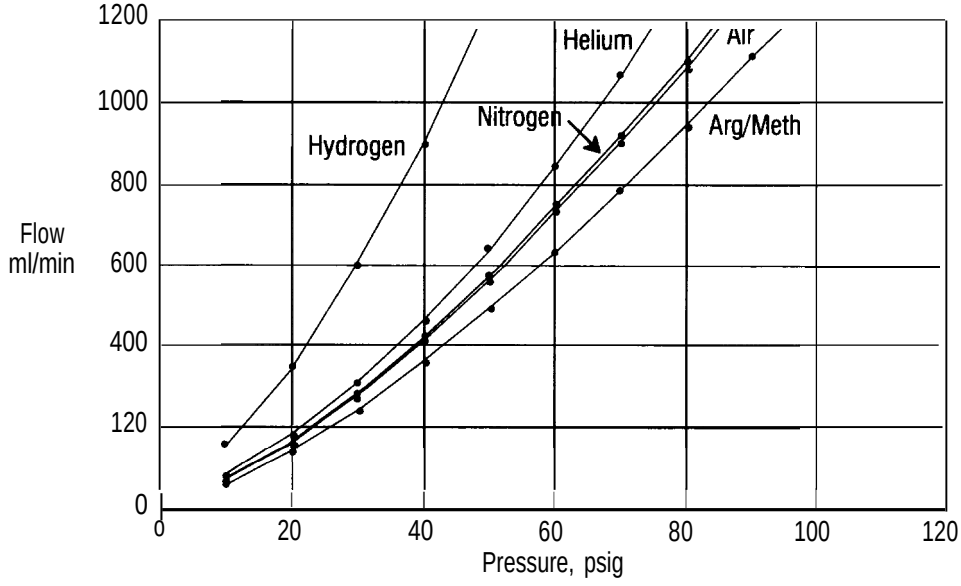


Flow Restrictor Data 19234-60600

Auxiliary EPC Restrictor Kit

19231-60610 Brown Dot
Computed nominal values at ambient temperature of 21°C and pressure of 14.56 psia

Pressure (kPa)	Pressure (psig)	Helium Flow (ml/min)	Nitrogen Flow (ml/min)	Hydrogen Flow (ml/min)	Air Flow (ml/min)	Argon/Meth Flow (ml/min)
69.0	10	**76**	61	150	**65**	**55**
137.9	20	**174**	161	344	**157**	**134**
206.8	30	**302**	279	596	**273**	**235**
275.8	40	**457**	418	896	**410**	**352**
344.7	50	**634**	571	1243	**561**	**485**
413.7	60	**838**	740	1634	**726**	**627**
482.6	70	**1063**	915	2035	**900**	**782**
551.6	80	**1310**	1101	2456	**1084**	**945**
620.5	90	**1580**	1297	2918	**1278**	**1110**
689.5	100	**1873**			**1470**	**1270**



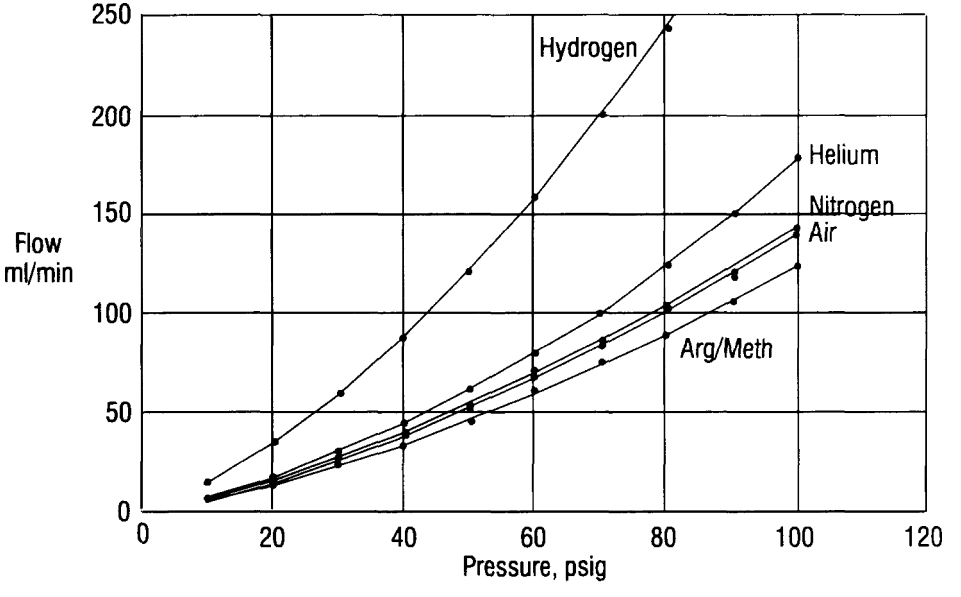
Flow Restrictor Data 19231-60610

Setting pressure using auxiliary electronic pressure control

Auxiliary EPC Restrictor Kit

19243-60540 Green and Red Dot
Computed nominal values at ambient temperature of 21°C and pressure of 14.56 psia

Pressure (kPa)	Pressure (psig)	Helium Flow (ml/min)	Nitrogen Flow (ml/min)	Hydrogen Flow (ml/min)	Air Flow (ml/min)	Argon/Meth Flow (ml/min)
69.0	10	7.2	6.4	14.7	6.2	5.7
137.9	20	17	15	35	15	13
206.8	30	29	26	59	25	22
275.8	40	44	39	88	38	33
344.7	50	61	53	122	52	46
413.7	60	80	69	159	67	59
482.6	70	100	86	200	84	74
551.6	80	124	104	243	101	89
620.5	90	150	123	290	120	106
689.5	100	178	143	339	139	123

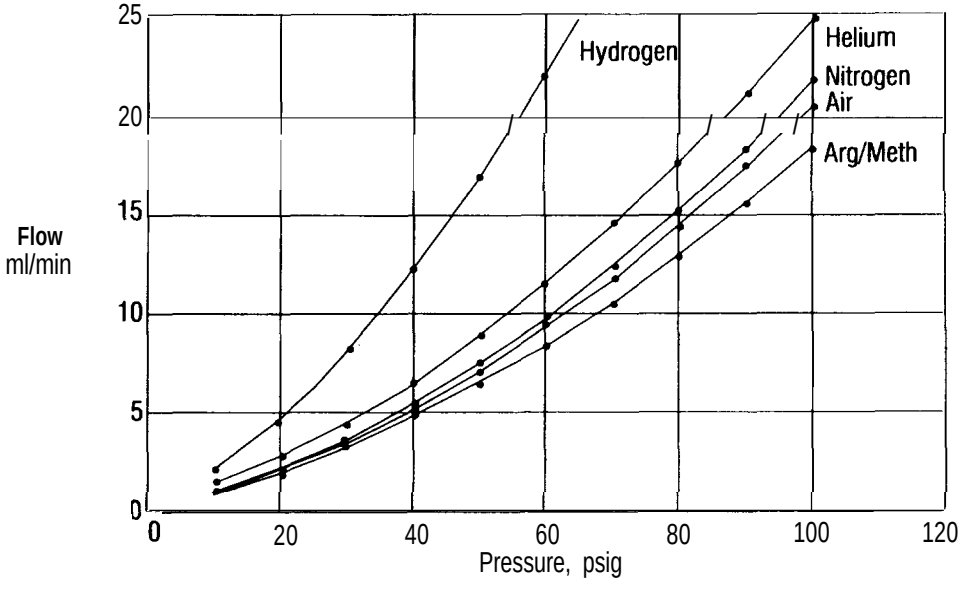


Flow Restrictor Data 19243-60540

Auxiliary EPC Restrictor Kit

19234-60660 Blue Dot
Computed nominal values at ambient temperature of 21°C and pressure of 14.56 psia

Pressure (kPa)	Pressure (psig)	Helium Flow (ml/min)	Nitrogen Flow (ml/min)	Hydrogen Flow (ml/min)	Air Flow (ml/min)	Argon/Meth Flow (ml/min)
69.0	10	1.3	1.0	2.0	0.9	0.9
137.9	20	2.7	2.1	4.6	2.0	1.9
206.8	30	4.4	3.6	8.1	3.4	3.2
275.8	40	6.4	5.4	12.3	5.0	4.8
344.7	50	8.8	7.4	16.9	7.0	6.4
413.7	60	11.5	9.7	22.2	9.2	8.4
482.6	70	14.5	12.3	27.9	11.7	10.5
551.6	80	17.7	15.2	34.5	14.5	12.9
620.5	90	21.2	18.3	41.5	17.5	15.5
689.5	100	24.9	22.0	49.7	20.6	18.4



Flow Restrictor Data 19234-60660

Using electronic pressure control to control gas flow

With EPC you can also control flow by setting the pressure. The following procedures will show you how to:

. Access the flow parameter displays

● Select the gas type

. Set the column diameter

. Set the column length

. Use the vacuum compensation mode

. Set constant mode for inlets

● **Set** mass flow rate for inlets

Pressure is the parameter controlled and measured with the EPC system; however, the corresponding column outlet flow rate and average linear velocity are also calculated and displayed. Entries can be made in terms of velocity from which the system calculates the required pressure and enters this setpoint

The following example shows the relationship between pressure and flow for EPC systems. In the first table, pressure is constant and the flow changes with temperature. In the second table, flow is constant and pressure changes with temperature.

Constant Pressure with Changing Flow

Temperature (°C)	**50**	**100**	**150**	**200**	**250**	**300**
Flow (ml/min)	3.6	2.8	2.3	1.9	1.6	1.3
Pressure (psi)	15	15	15	15	15	15
Linear Velocity (cm/see)	51.1	46.3	42.4	39.1	36.2	33.7

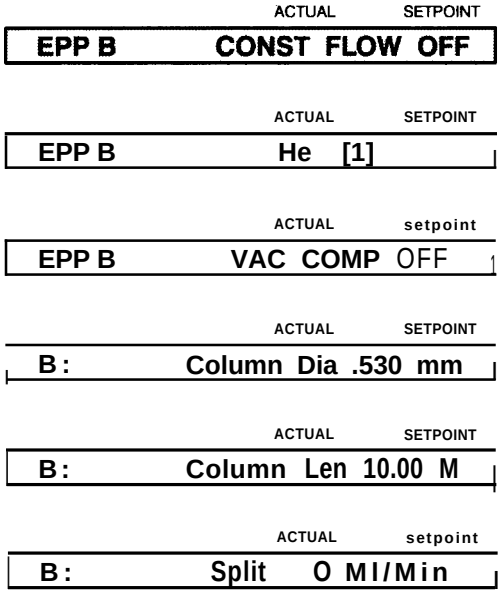
Constant Flow with Changing Pressure

Temperature (°C)	50	100	150	200	250	300
Flow (ml/min)	3.6	3.6	3.6	3.6	3.6	3.6
Pressure (psi)	15	18	21.9	24	27.1	30.2
Linear Velocity (cm/see)	51.1	55.1	58.3	60.9	63.1	65.0

Accessing the flow parameter displays

Use the following steps to access and scroll through the GC flow parameters displays.

1. Press: gold FLOW to access the flow parameters displays.
2. Continue to press: FLOW to scroll through the flow parameter displays. The displays may be in a different sequence depending on your configuration.



Use this display to turn the constant flow mode on or off.

Use this display to change the gas type.

Use this display to turn the vacuum compensation mode on or off.

Use this display to set the column diameter

Use this display to set the column length.

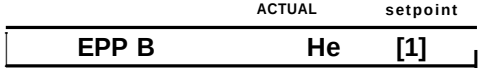
Use this display to set the mass flow rate.

Flow Parameter Displays

Selecting the gas type

You will need to selector verify the gas type you are using for EPC applications. To select the gas type:

1. Press: gold FLOW to access the flow parameters display.
2. Continue to press: FLOW until the gas type appears on the GC display.

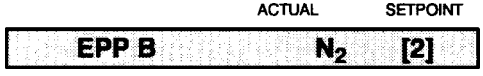


The GC display now looks like this.

3. Press the number corresponding to the gas type you want to use. The chart below lists the gas types available:

	Gas Type
1	Helium
2	Nitrogen
3	Hydrogen
4	Argon/Methane

The number and gas you select will appear under the" setpoint" column on the GC display:



The GC display now looks like this.

Setting the column diameter

To set the column diameter:

1. Press: gold FLOW to access the flow parameters display.
2. Continue to press: FLOW until you see the column diameter display.

	ACTUAL	SETPOINT
B Column DIA	.XXX m m	

The GC display looks like this.

3. Enter the column diameter in µ (such as 200 µ, 320 µ, 530 µ). The example below shows the column diameter for a 530 µ column.

Press: 5 3 0 ENTER *Sets the column diameter to 530 µ.*

	ACTUAI	SETPOINT
B: Column Dia	.530 mm	

The GC display looks like this.

Setting the column length

If you do not know the exact column length or if you are using a packed column, follow the steps described in *Determining the corrected column length* later in this chapter. To set the known column length in meters:

1. Press: gold FLOW to access the flow parameters display.
2. Continue to press: FLOW until you see the column length display.

	ACTUAL	SETPOINT
B: Column Len XX.XXM		

The GC display looks like this.

3. Enter the column length in meters.

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

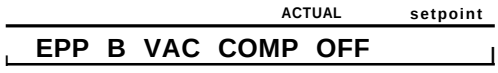
	ACTUAL	SETPOINT
B: Column Len 25.00M		

The GC display looks like this.

Using vacuum compensation mode

Use vacuum compensation mode when you are using a mass spectrometer to correct for the column outlet pressure. Using the vacuum compensation mode ensures that the constant flow mode, calculated column flow, and average linear velocity are correct.

1. Press: gold FLOW to access the flow parameters display.
2. Continue to press: FLOW until you see the vacuum compensation display.



The GC display looks like this.

3. Use one of the following steps to turn the vacuum compensation mode on or off:

   a. Press: ON to turn vacuum compensation mode on.

      After you select vacuum compensation, set the desired pressure. For more information on setting the inlet pressure, see *Setting inlet pressure using electronic pressure control* earlier in this chapter.

   b. Press: OFF to turn vacuum compensation mode off.

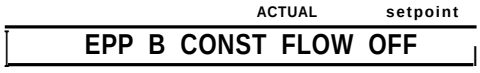
Using constant flow mode for inlets

Before setting the constant flow mode for inlets, you must first select the gas type. See *Selecting the gas type* earlier in this chapter for more information.

Note: The initial pressure changes when you turn the constant flow mode on or off. Enter the desired initial pressure after selecting constant flow mode. Also, make sure that the oven is equilibrated to the initial temperature. If you set the initial pressure before the oven reaches the initial oven temperature, the flow will be incorrect.

To use constant mass flow mode:

1. Press: gold FLOW to access the flow parameters display.
2. Continue to press: FLOW until you see the constant flow display.



3. Use one of the following steps to turn the constant flow mode on or off:

   a. Press: ON to turn the constant flow mode on.

      When you select on, you can set an initial pressure (at oven initial temperature) and the GC maintains the initial flow throughout the run by adjusting pressure continuously and automatically.

   b. Press: OFF to turn the constant flow mode off.

      You must select off if you want to create independent pressure programs.

Setting mass flow rate for inlets

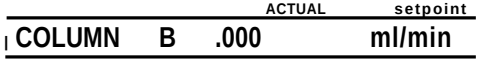
When a pressure is set, the mass flow rate is displayed. Entering a new mass flow value sets a new pressure automatically to produce the flow value entered.

1. Enter the correct gas type, column diameter, and column length. For more information on setting the correct column parameters, see the appropriate sections earlier in this chapter.

2. Use the following steps to set the inlet B mass flow rate to 10 ml/min:

   a. Press: CLEAR .

   b. Continue to press: FLOW until you see the mass flow control display.



The GC display looks like this.

   c. Press: 1 0 ENTER to set the inlet B flow to 10 ml/min.

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

Setting inlet flow programs

You can use.EPC to set flow programs indirectly by setting an inlet pressure program. The following example shows how to obtain the pressure values necessary to set a pressure program that results in the desired flow programs.

1. Enter the correct gas type, column diameter, and column length. For more information on setting the correct column parameters, see the appropriate sections earlier in this chapter.

1. Press: CLEAR .

2. Continue to press: FLOW until you see the mass flow control display.

	ACTUAL	setpoint
COLUMN B	.XXX	ml/min

The GC display looks like this.

3. Press: 4 ENTER to set the inlet B initial flow to 4 ml/min.

	ACTUAL	SETPOINT
COLUMN B	4.0	ml/min

The GC display looks like this.

4. Press: gold INJ B TEMP .

	ACTUAL	SETPOINT
EPP B	10.0	10.0

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

5. Press: FLOW repeatedly until you *see* the mass flow control display.

	ACTUAL	SETPOINT
COLUMN B	4.0	ml/min

The GC display looks like this.

6. Press: 7 ENTER to set the inlet B flow to 7 ml/min.

	ACTUAL	SETPOINT
COLUMN B	7.0	ml/min

The GC display looks like this.

7. Press: gold INJ B TEMP .

	ACTUAL	SETPOINT
EPP B	*14.3*	*14.3*

*The GC display looks like this.****The value shown will be the inlet pressure setting needed to obtain the flow you entered.***

Use the pressure values obtained from this procedure for setting a pressure program. Enter the initial time, ramp rate, and final time as part of setting the pressure program (see *Setting pressure programs* earlier in this chapter). You will also need to change the oven temperature if it will change during the pressure program.

Setting the average linear velocity

You can use EPC to set the calculated average linear velocity for inlets. For example, when you set a pressure, the average linear velocity is displayed (while monitoring the average linear velocity). The following sections will show you how to:

. Understand average linear velocity

● Calculate the outlet flow

. Set the average linear velocity

. Calculate the outlet linear velocity

. Calculate the actual average linear velocity

Understanding average linear velocity

The average linear velocity is calculated and measured at oven temperature, rather than at ambient temperature. It is an average value because velocity varies continually along the length of the column (due to the pressure drop and the compressibility of the carrier gas).

To compare experimental values with those displayed by the system, measure the outlet flow and compare it to the calculated outlet flow. Then measure the average linear velocity and compare it to the velocity displayed. You need to consider the corrections for both temperature and compressibility if you use the average linear velocity (unretained peak time) measurements to calculate flow. You can calculate an approximate value for flow without correcting for compressibility, but it may differ significantly from the flow value displayed by the system as the pressure drop increases. A more detailed discussion of these calculations and pressure versus flow relationships is given in Appendix A, *Pressure versus flow relationships for inlet and auxiliary electronic pressure control.*

Calculating outlet flow

Outlet flow is the gas flow out of the column. It is measured in ml/min and corresponds to the measurements made with a flow meter at the detector outlet. The gas is measured at ambient conditions; however, the outlet flow displayed is calculated using 25 °C and 1 atmosphere pressure (14.7 psi) as reference conditions.

All flow calculations are based on the ideal gas law.

$$PV = nRT$$

where T = temperature in K
P = absolute pressure

Ideal Gas Law

Setting the average linear velocity

Use the following steps to set the approximate column B average linear velocity to 100 cm/sec.

1. Enter the correct gas type, column diameter, and column length. For more information on setting the correct column parameters, see the appropriate sections earlier in this chapter.
2. Use the following steps to set the column B average linear velocity to 100 cm/see:

   a. Press: CLEAR .

   b. Continue to press: FLOW until you see the average linear velocity display

	ACTUAL	SETPOINT
COLUMN B	97.5 Cm/Sec	

The GC display looks like this.

The value displayed (97.5 cm/sec) is the computed average linear velocity ($\bar{u}$) for the correct pressure, temperature, and gas type.

c. Press: 1 0 0 ENTER . *Sets inlet B linear velocity to 100 cm/sec.*

Note: Inlet B pressure will change to a value needed to produce 100 cm/sec at the current oven temperature. Inlet B flow also corresponds to that pressure.

Determining the corrected column length

Packed column considerations

. Calculations for the pressure versus flow relationship with EPC apply to flow through open tubular columns.

  Measure flow versus pressure to determine the relationship for a packed column and to check for changes as a column is used.

. Operating pressure may reach the 100 psi limit for longer columns or higher temperatures.

. EPC offers many advantages over mass flow controllers, including:

  – Precision and reproducibility of setpoints

  – Rapid adjustment to change in settings during downstream operation

  – Pressure programming capability for reduced run times

To set or display the mass column flow for a packed column, you must first calculate the corrected column length and diameter for an equivalent open tubular column.

Capillary column considerations

Although column specifications show the nominal length of the column, not all columns are manufactured exactly to the nominal specifications. Also, previously used columns are shorter than" their specifications if the ends were cut off to remove contaminants.

To compensate for both packed and capillary column considerations, use the following procedure to determine the corrected column length and diameter.

Note: Before setting the mass flow rate, enter the correct gas type, column diameter, and column length. For more information on setting the correct column parameters, see the appropriate sections earlier in this chapter.

1. Use the following steps to set the column B average linear velocity:

   a. Press: CLEAR .

   b. Press: FLOW repeatedly until the display reads:

	ACTUAL	SETPOINT
COLUMN b	97.5	Cm/Sec

   The value displayed (97.5 cm/see) is the computed average linear velocity ($\bar{u}$) for the estimated length (10 M) column.

2. Inject an unretained component into the GC and determine its retention time in minutes. This is t_0 Actual in the following equation.

3. Use the following formula to calculate the corrected column length:

$$L_{corrected} = \left(\frac{t_0 \text{ Actual} \times \bar{u}}{1.67} \right)$$ where:

$L_{corrected}$	= corrected column length in meters
t_0 Actual	= retention time of unretained component in minutes
$\bar{u}$	= average linear velocity in cm/sec
1.67	= (cm to M) 60 min corrections

4. Press: gold FLOW to access the flow parameters display.

5. Continue to press: FLOW until you see the column length display.

	ACTUAL	SETPOINT
B: Column Len	XX.XXM	

The GC display looks like this.

6. Enter the value calculated from the above formula as the corrected column length.

   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. See Chapter 4, *Using the internal stopwatch* for more details.

Optimizing splitless injection using electronic pressure control

The inlet carrier gas pressure at the time of injection can affect the transfer of sample to the column dramatically. With low inlet carrier gas pressure, the column flow rate is slower so the sample stays in the inlet longer. Because of this, the sample has more time to expand. In addition, low inlet carrier gas pressure results in lower inlet pressure and a larger sample expansion volume. Conversely, higher flows (or higher pressures) at the time of injection cause the sample to be swept into the column more rapidly, thus reducing the expansion volume.

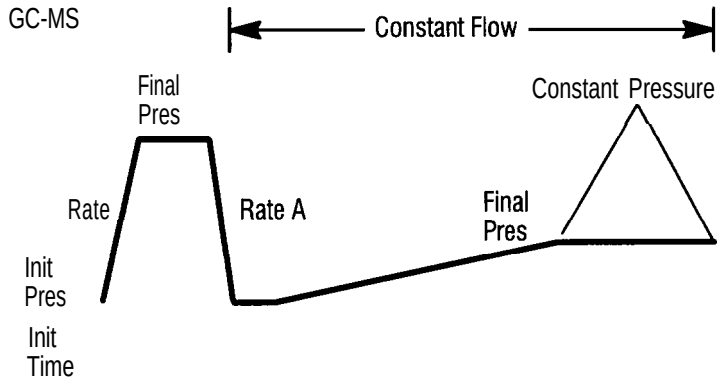
Because the inlet pressure can be programmed up or down, it is possible to initiate a run with a high flow rate and then program the flow downward to a value that is optimal for the chromatographic separation.

splitless injection volumes are usually limited to 1 to 2 µl of sample, but this is highly dependent upon factors such as the inlet temperature, column flow rate, solvent molecular weight, solvent boiling point, liner volume, column type, and retention gap use. With larger injections, poor sample transfer can result in sample losses and molecular weight discrimination.

A slightly modified pressure programming technique is appropriate for GC-MS systems. The program starts at a low initial pressure, then ramps up at the beginning of the GC run, and ramps down again after the sample has been transferred to the column.

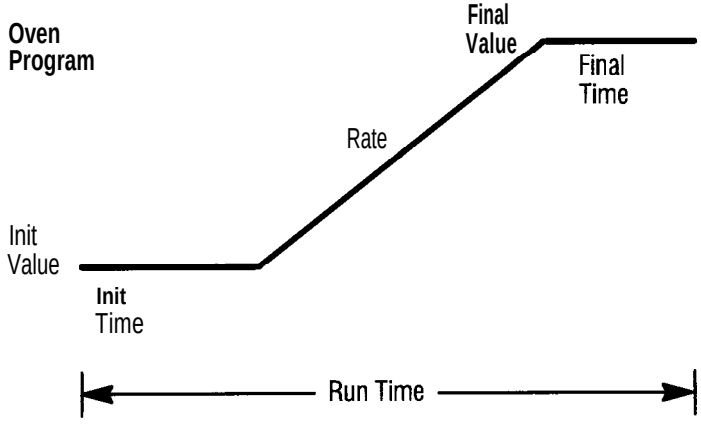
An example of rapid pressure programming for a GC-MS system is shown below.

Rapid Pressure Program



Init Pres	10
Init Time	0
Rate	**99**
Final Pres	40
Final Time	0.25
Rate A	99
Final Pres A	10
Final Time	0

Single-Ramp Oven Temperature Program



Init Value	100
Init Time	2
Rate	10
Final Value	200
Final Time	1

Example Setpoints of Rapid Pressure Programming

Operating the gas saver application for the split/splitless inlet

What is the gas saver application?

The gas saver application is one use of the auxiliary EPC system. It allows you to control the split vent flow of a split/splitless inlet by controlling supply pressure to the inlet. By controlling the pressure, you can reduce the total flow rate during nonproductive run times and laboratory off-hours, which will reduce your GC operating costs. The gas saver is particularly beneficial for users of expensive carrier gas and for capillary inlet systems used with high split flows. When properly programmed for a capillary inlet system, constant column head pressure and column flow rate are maintained while the excess split flow is reduced.

What are the required settings for operation?

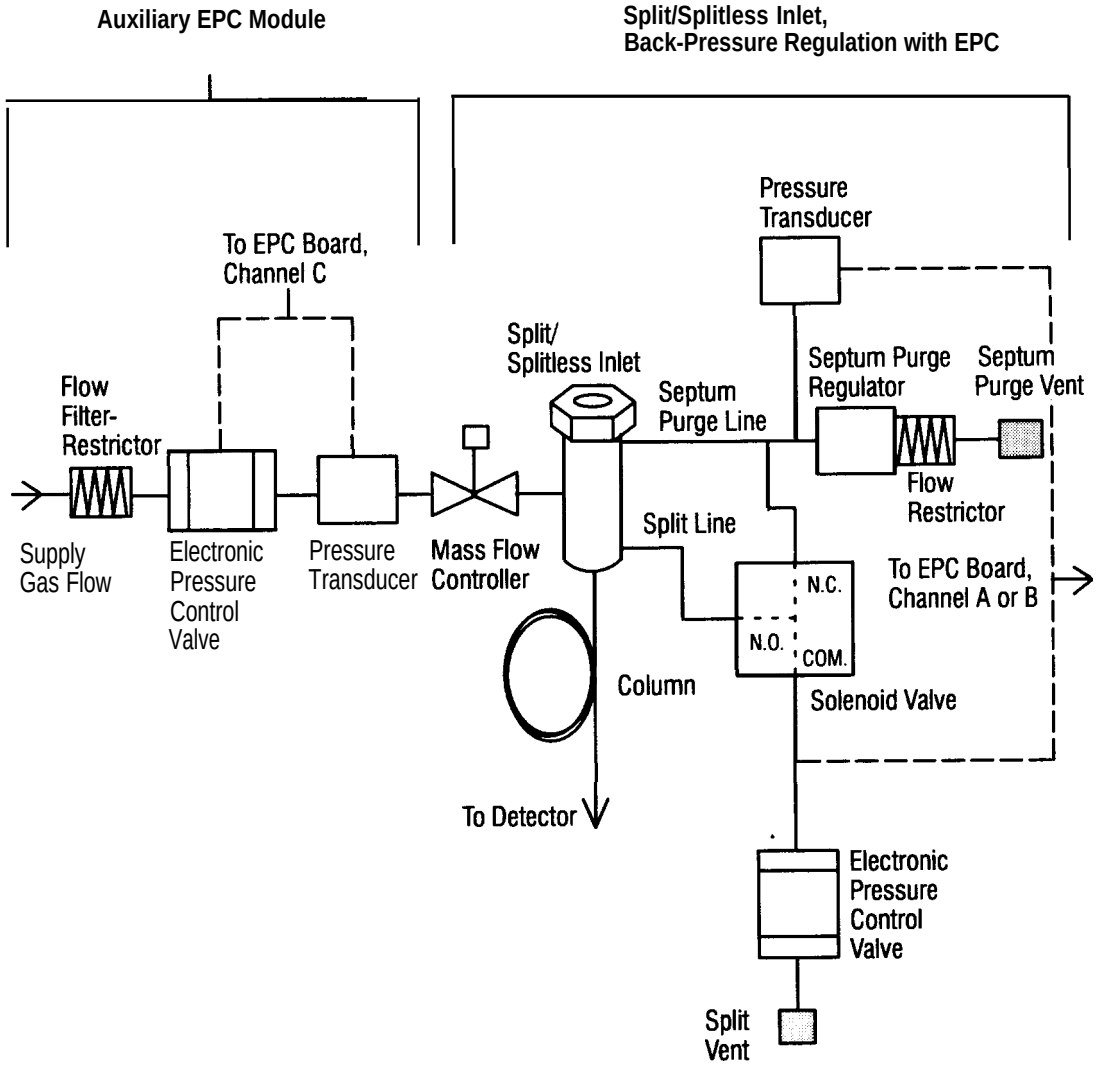
When you operate the gas saver, you must set the auxiliary pressure at least 5 to 10 psi greater than the inlet head pressure to ensure that column pressure and flow are maintained. To determine the minimum auxiliary setting, detect the maximum pressure of the programmed run and add 5 to 10 psi. If you are not using constant flow mode, the column pressure drop increases and the column flow decreases. If you are using constant flow mode, the pressure will increase during temperature programming. In general, the operating constraints for the gas saver application are as follows:

. Operate at least 5 to 10 psi above column head pressure (some minimal split vent flow is required).

. Operate at least 10 psi above the supply line pressure (or as maintained by the system).

. Use the GC displays and warnings when configuring.

How is the gas saver application configured?



How does the gas saver application operate?

When an EPC module is used for gas supply to the split/splitless inlet (gas saver configuration), restriction is due mainly to the mass flow controller. You can vary the restriction by changing the setting of the mass flow controller.

To operate in gas saver mode, you need to enter the gas type, column length, column diameter, column pressure, and split flow rate. You will also need to calibrate the flow versus the pressure, set the mass flow controller to the desired range, and determine the column head pressure. Be sure that all the hardware is installed properly and that all gases are plumbed.

Use this procedure to determine the settings that will yield the most gas savings for your system:

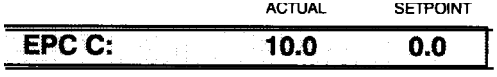
Zero the channel

1. Check your system for leaks.
2. Zero your channel if ambient conditions have changed significantly:
   a. Make sure that all heated zones are cool.
   b. Set the auxiliary and inlet pressures to zero.
   c. Remove the septum nut to repressurize the system completely. The example below shows the display for auxiliary EPC channel C.

      Note: Setting the inlet pressure to 0.0 may not repressurize the channel completed. You may also need to bleed off at the l/8-in Swagelok fitting.

   1. Press:

Sets the channel C pressure to 0.0.



The GC display looks like this.

e. Press: gold 4 ENTER *10* ENTER

where 10 is the zero offset value labeled "actual" on the GC display.

Enter the carrier gas pressure

1. Enter the carrier gas supply pressure at the keyboard. A typical pressure is 50 to 60 psi.
   For example:

   a. Press:

gold A 5 0 ENTER — *Sets the channel C pressure to 50.0.*

	ACTUAL	SETPOINT
EPC C:	50.0	50.0

The GC display looks like this.

2. Turn the mass flow controller on the front of the flow panels until you measure 80 to 100 ml/min (or the desired flow) with the bubble flow meter.

   Note: You must wait 1 to 2 minutes after changing pressure or mass flow controller settings to allow the system to stabilize before measuring flows or making an injection.

Enter the column parameters

1. At the keyboard, press: gold FLOW to access the flow parameters table.
2. Press: FLOW to scroll through the table. Enter your values for the following:

   - Constant flow (on or off)
   - Gas type
   - Vacuum compensation (on or off)
   - Column diameter

. Column length

- Split flow

For more information on setting these parameters, see the appropriate sections earlier in this chapter.

3. To verify the column flow, measure the flow out of the detector with a bubble flow meter.

Set the system to operating conditions

Use the flow restrictor tables described in *Setting pressure using auxiliary electronic pressure control* earlier in this chapter to select the makeup gas pressure that will yield the flow you need. For example, the following steps describe how to set the pressure to get 30 ml/min makeup gas flow.

1. Set the inlet pressure to 10 psi (if it is not set already).
2. Set the makeup gas at the keyboard to get to approximately 30 ml/min.
3. Start to heat any heated zones that are not already heated.

WARNING **Heat** the detector before you continue.

4. If you did not select constant flow, verify the flows after the zones reach their desired temperatures.
5. After the system reaches equilibrium, check and record the final pressure at the maximum operating temperature.

   Note: Record the final pressure so that you know what value to set for the reduced gas saver pressure, and then observe the pressure at the maximum operating temperature. If you want a higher temperature than listed in the operating manual, realize what that pressure will be when you reset it.

Set the system to off-hour conditions

If the final column pressure is 50 psi, set the carrier gas pressure to 65 psi. During off-hours, you can program the carrier gas down.

1. Program the desired oven temperature.
2. Set all detector and inlet flows and pressures to the desired levels.
3. Set the carrier gas pressure to a value 15 psi higher than the column head pressure.

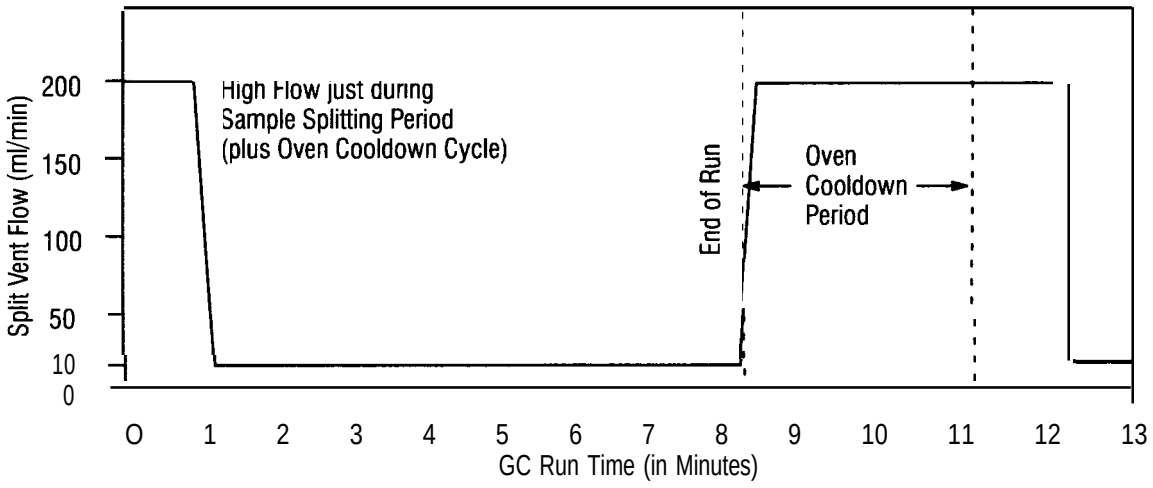
Recommended flow rates for inlet systems using the gas saver application

For a capillary split/splitless injection port, a combined flow rate out of the split vent and the purge vent of approximately 5–10 ml/min is recommended.

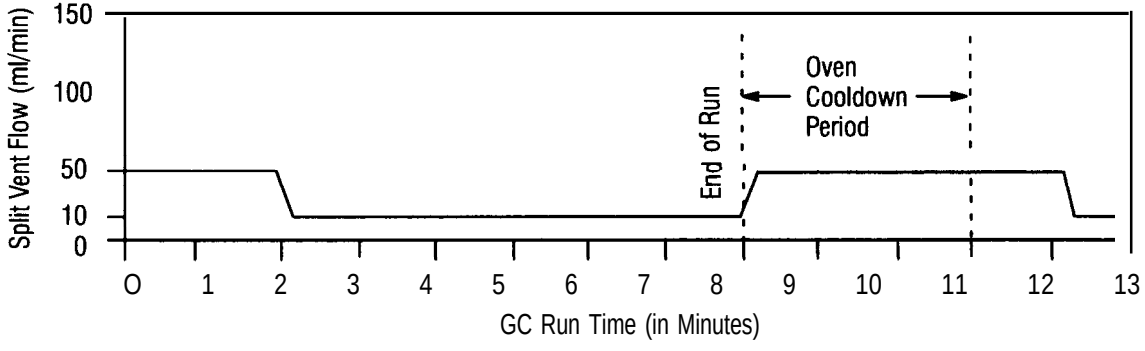
Additional benefits of the gas saver application

If desired, the makeup gas flow can also be reduced using the gas saver mode during off-hours to increase total gas savings. Plumb the gas saver outlet line into the detector manifold inlet fitting for makeup gas. Then set it using the same procedure as for a carrier gas.

With a capillary inlet, the gas saver can also be used as a quick, convenient way to set split ratio. The graphs below show examples:



Split Injection



splitless Injection

Using the external sampler interface

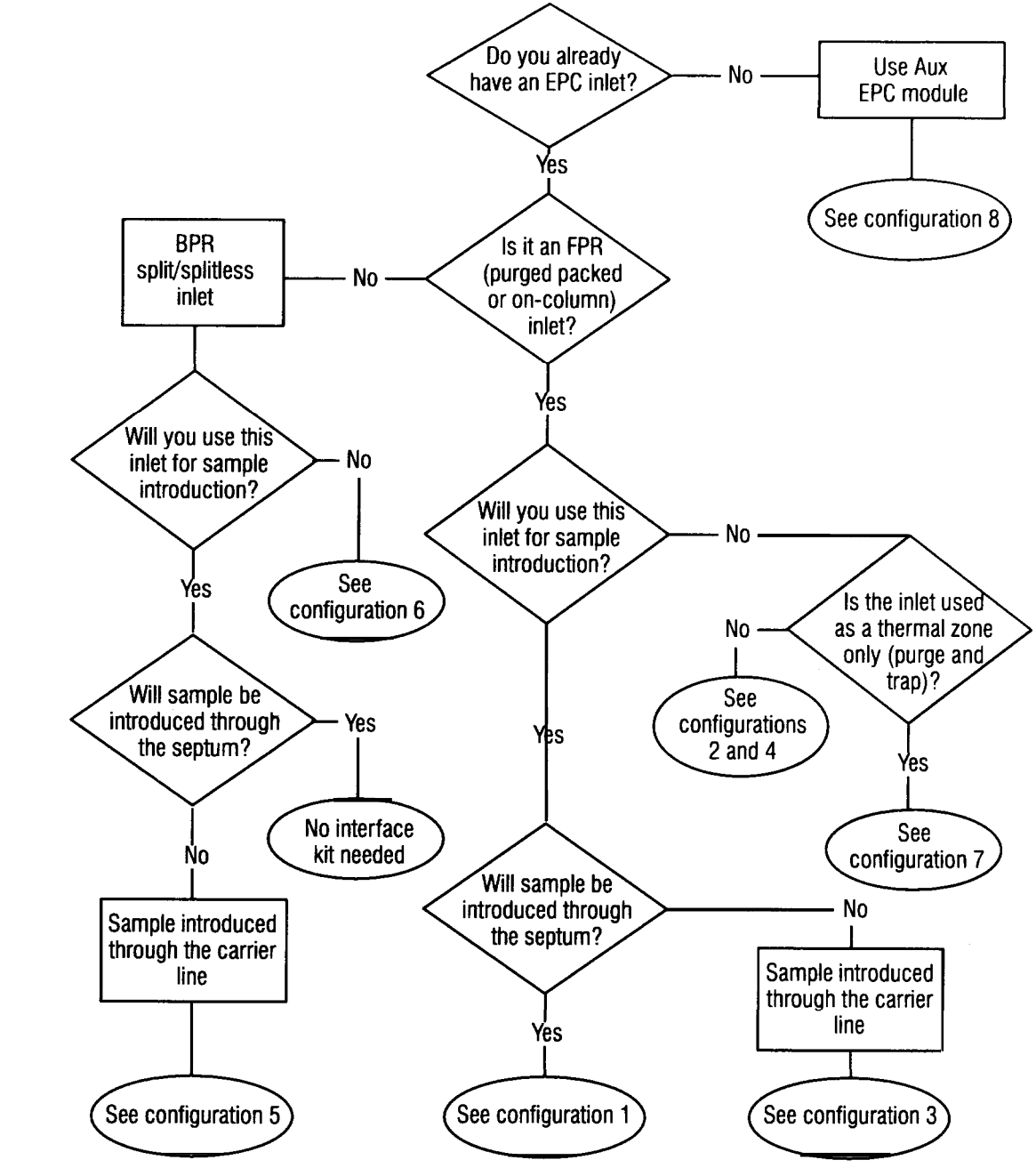
The external sampler interface kit (HP part number 19245-60990) enables you to use EPC with a sampling device other than a standard HP inlet. The conventional interfacing of external sampling devices is not possible when EPC inlets are used. The external sampler kit provides the hardware and diagrams for installing the kit onto forward-pressure controlled inlets (programmable cool on-column and purged packed). The kit also contains hardware for limited use of the back pressure-regulated split/splitless inlet. This hardware allows the system to sense the pressure at a different location inside the HP pneumatic system.

Which configuration should I use?

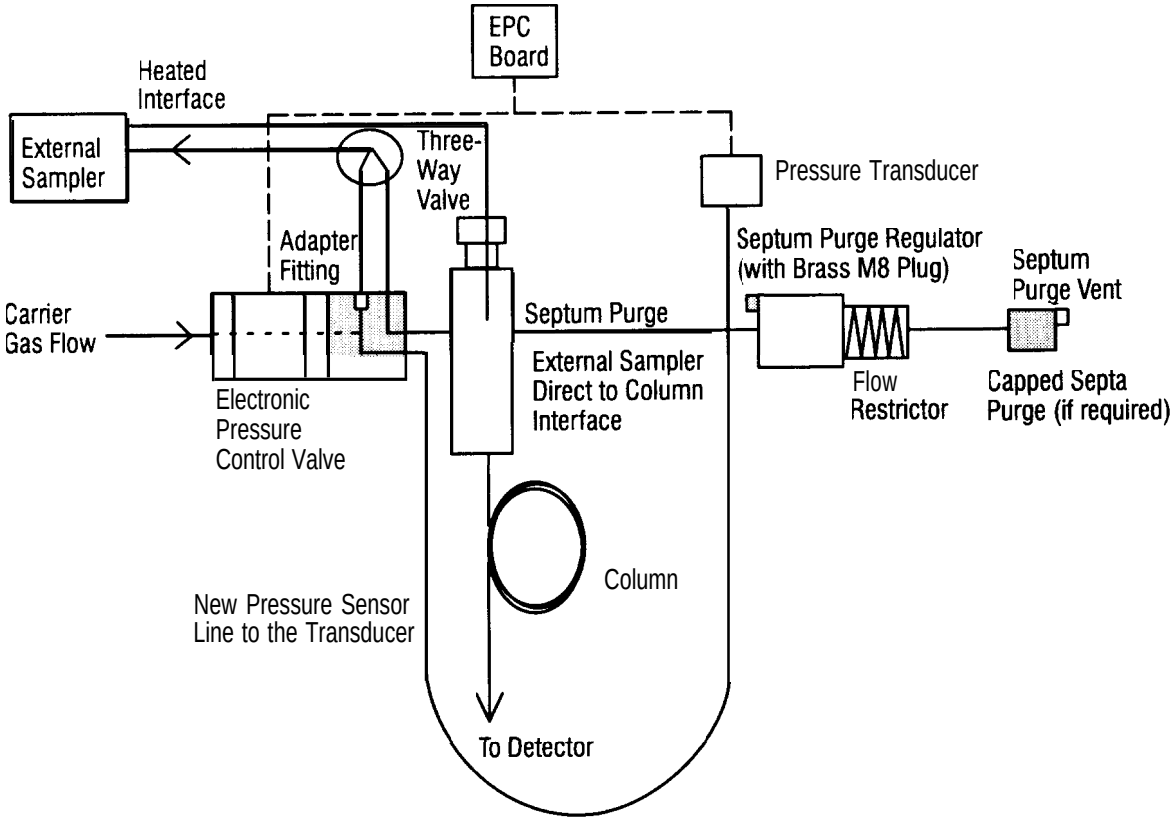
The external sampler interface is configured differently depending on the type of inlet you use. You will select one of several typical configurations of the external sampler interface depending on how you use your inlet. Use the following tables to determine the configuration you need. The diagrams shown in this section include the most commonly configured systems.

Inlet Use	External Sampler Interface Kit	Auxiliary Module
External Inlet	●	●
HP inlet used as a thermal zone	●	●
HP inlet used for injection	●	

	Forward-Pressure Regulation	Back-Pressure Regulation
Inlet Use	Front End Sampler for Purged Packed and On-Column Inlets	Front End Sampler Interface for split/splitless Inlets
External Interface	●	●
HP inlet used for sample introduction	●	●
HP inlet not used for sample introduction	●	●

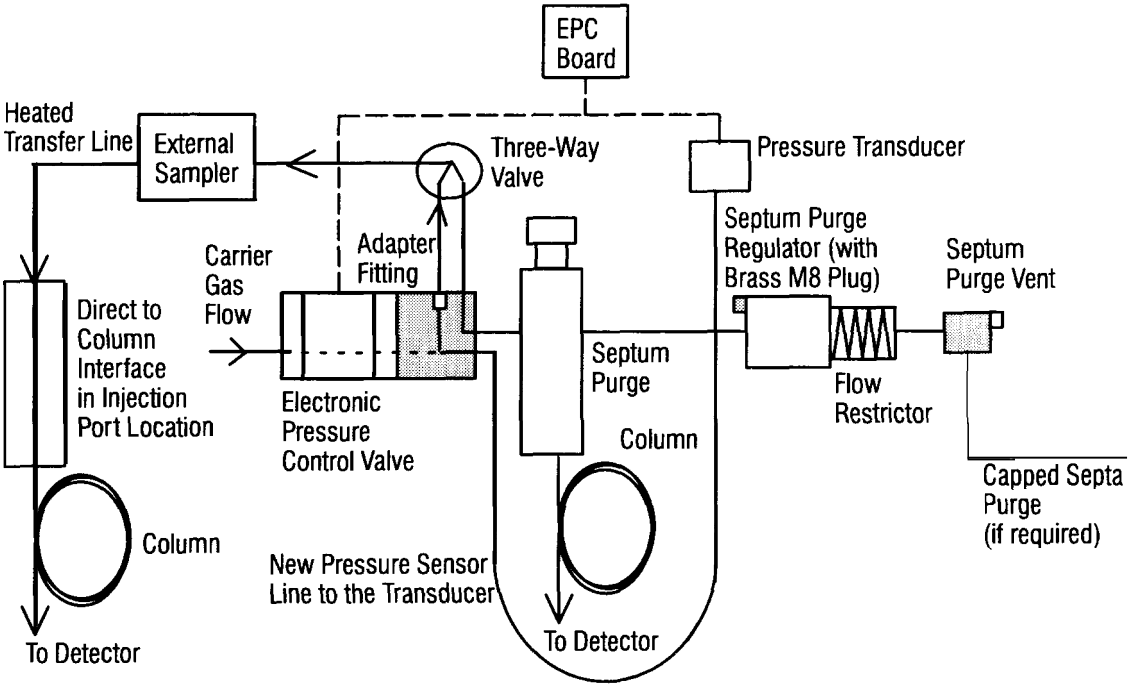


External Sampler Interface Configuration Decision Tree



Forward-pressure regulation for the septum-purged packed inlet EPC used with open tubular columns). External sampler interface (needle through septum) to HP inlet with forward-pressure control. A static headspace sampler can be interfaced in this way.

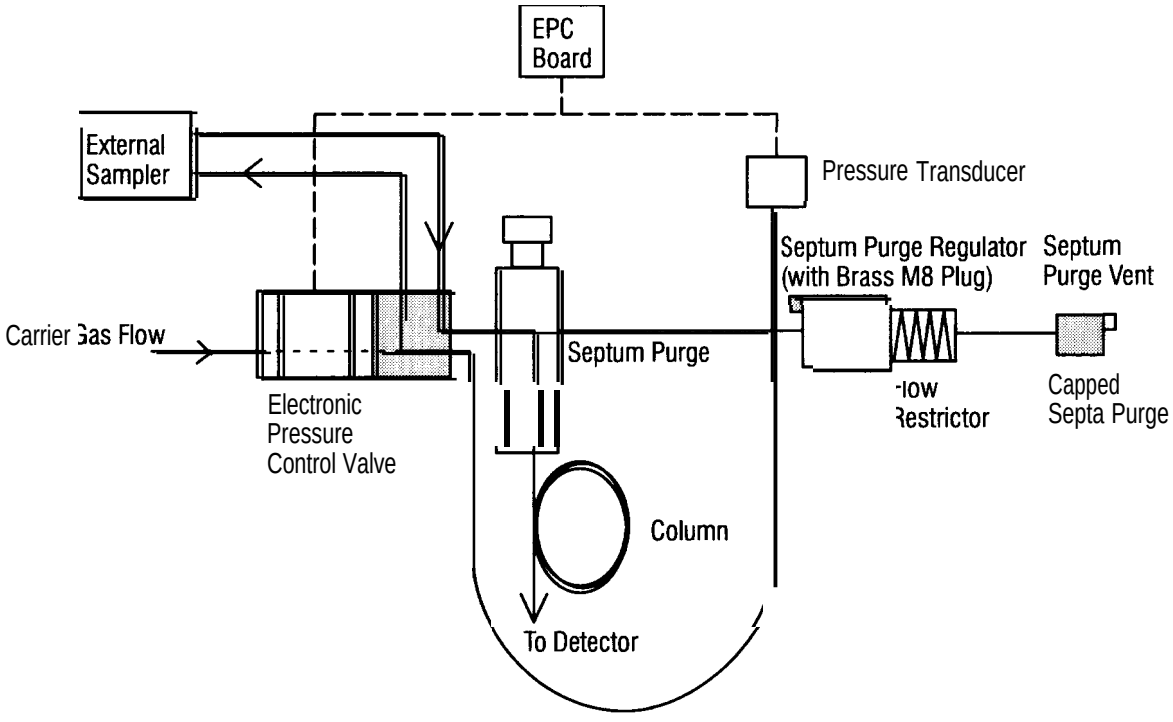
Configuration 1: External Sampler Interface to HP Inlet with FPR



Forward-pressure regulation for the on-column inlet and the septum-purged packed inlet (EPC used with open tubular columns). To External Sampler direct column interface or to HP inlet. Use the HP inlet with forward EPC.

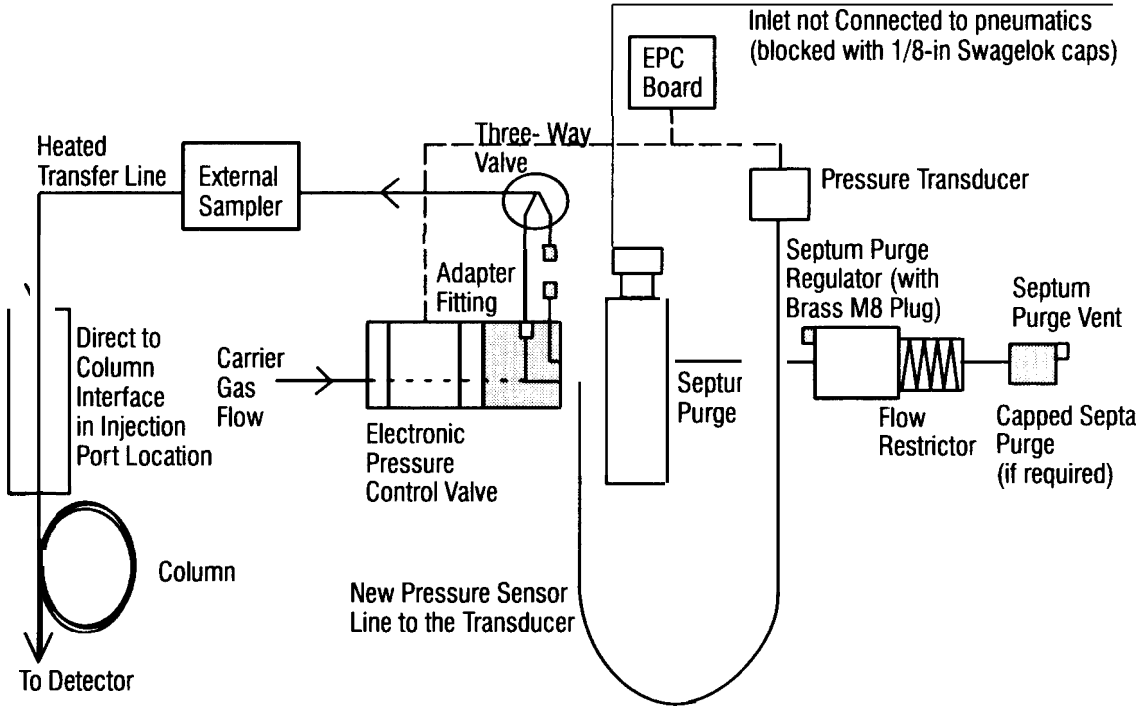
Configuration 2: External Sampler Interface to Column or to HP Inlet with FPR

The position of the three-way valve directs the EPC flow to the external sampler or to the HP inlet. This diagram shows the EPC flow directed to the external device.



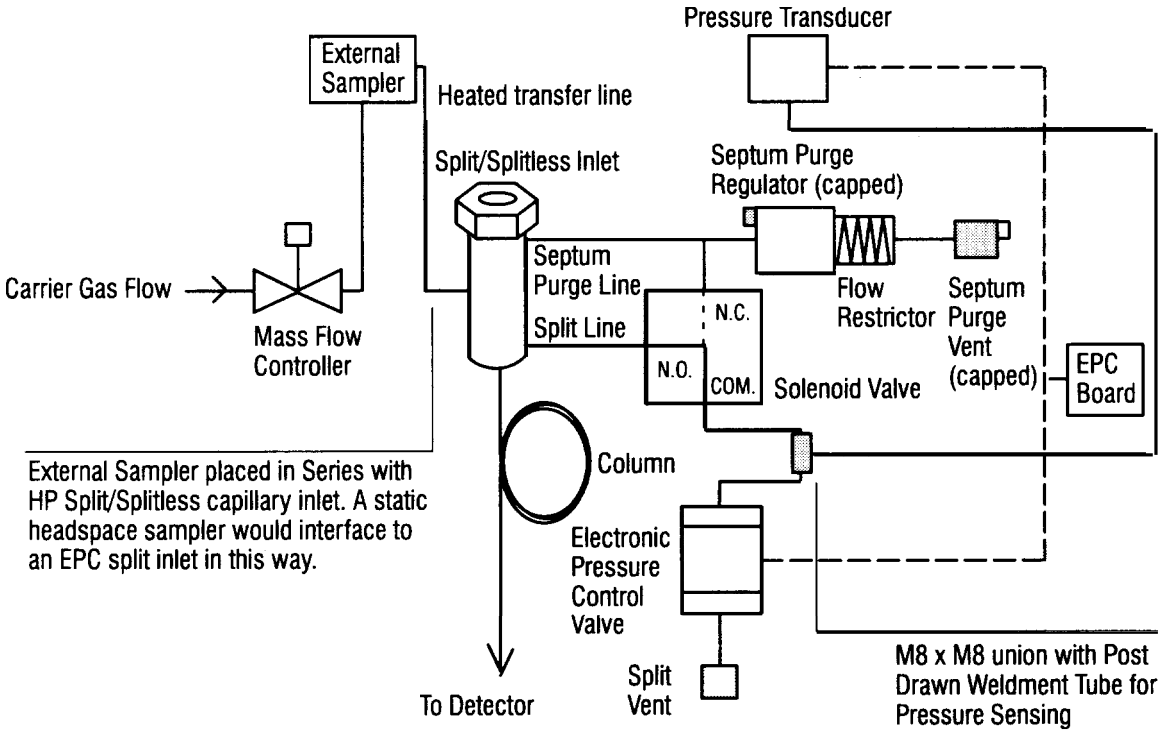
Forward-pressure regulation for the on-column inlet and the septum-purged packed inlet EPC used with open tubular columns). FromHP EPC pneumatics to external sampler and return to HP inlet. This configuration should not be used with compounds that have a retention index greater than 400-450.

Configuration 3: EPC to External Sampler to HP Inlet with FPR



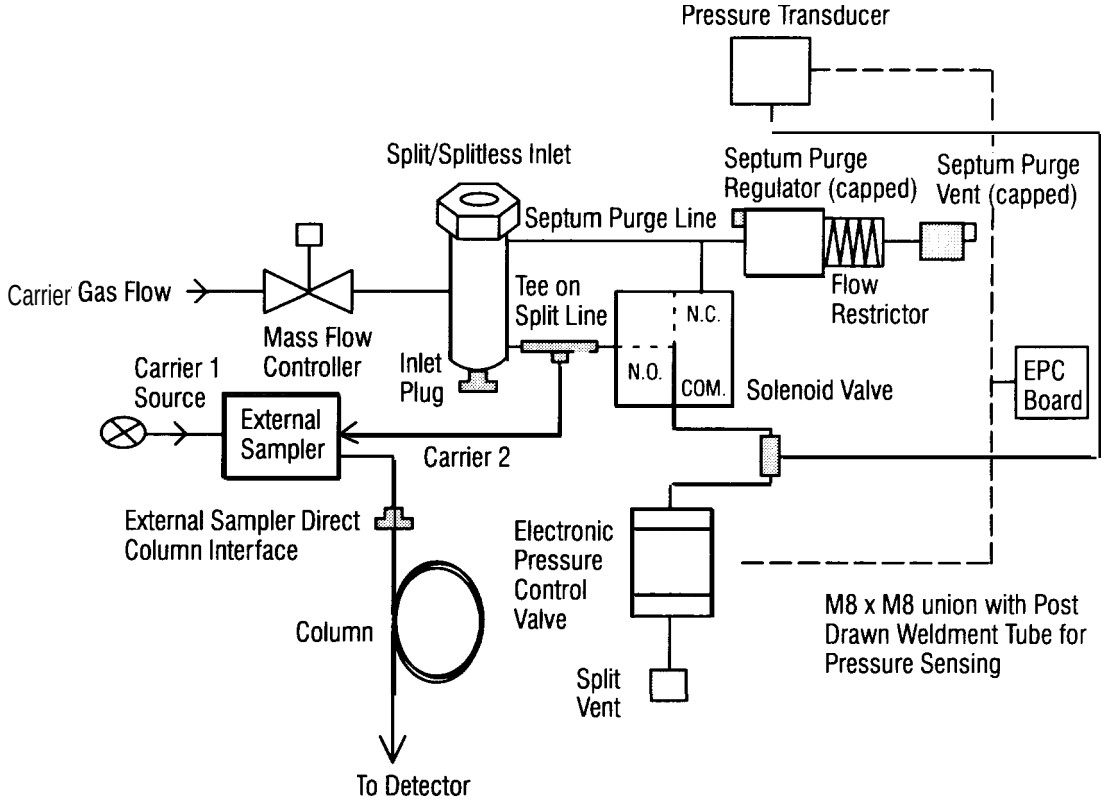
Forward-pressure regulation for the on-column inlet and the septum-purged packed inlet EPC used with open-tubular columns). To external sampler only. The HP pneumatics are used. The HP inlet is not functional.

Configuration 4: EPC to External Sampler



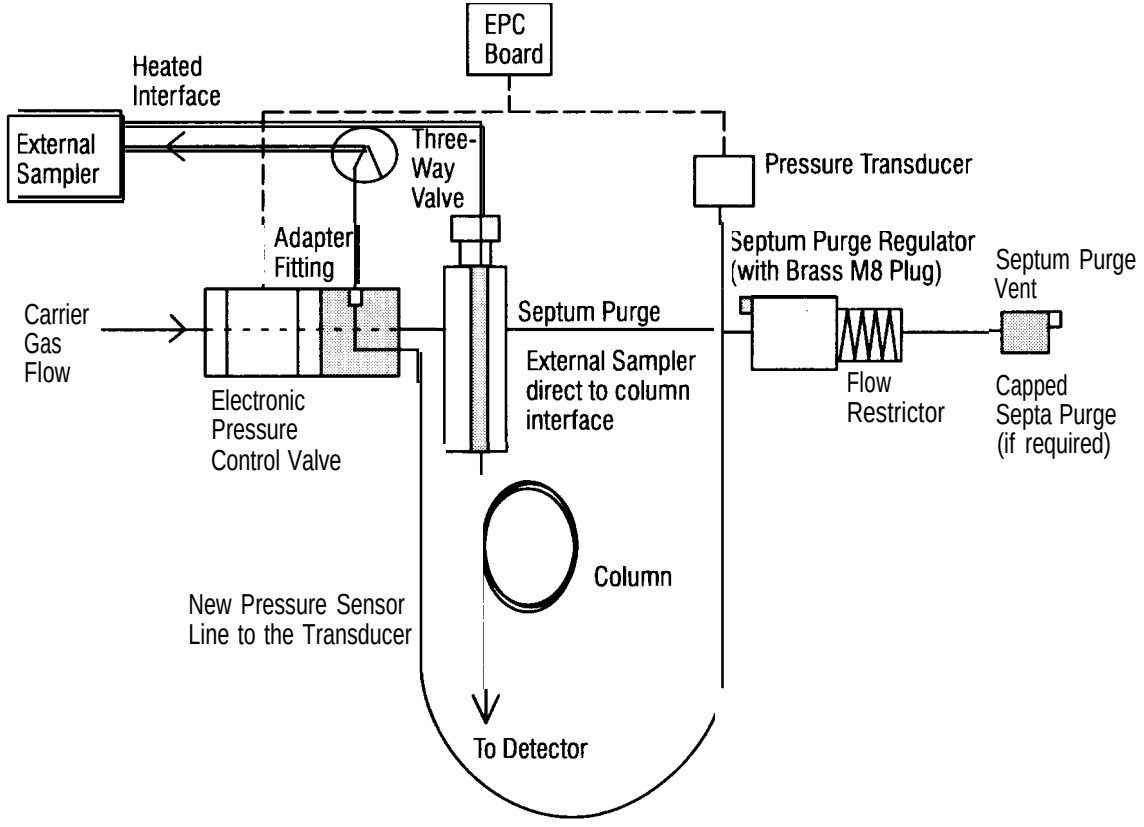
Back-pressure regulation for the split/splitless inlet. The external sampler is placed in the HP split/splitless capillary inlet flow system (in series). The external sampler transfer line was interfaced (cutting l/16-in. od tubing is required) close to inlet carrier in line. Septa purge is capped to prevent sample losses. The pressure sensing takes place just before the EPC valve.

Configuration 5: Back Pressure Regulated EPC with Inlet



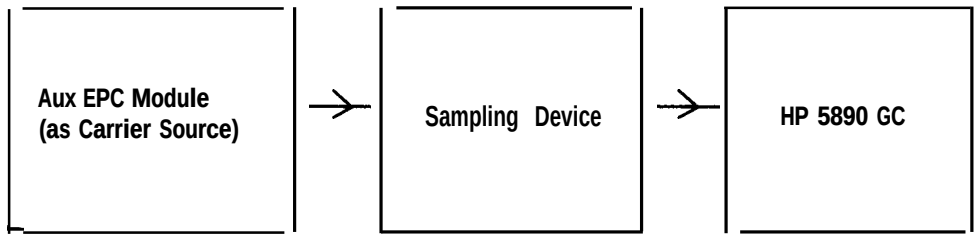
Back-pressure regulation for the split/splitless inlet. External sampler using the HP inlet back-pressure regulation of column. HP inlet not used, The external sampler has its own direct column interface in the unused inlet opening.

Configuration 6: Back Pressure Regulated EPC with No Inlet



Forward-pressure regulation for the septum-purged packed inlet EPC used with open tubular columns). External sampler interface direct to column with HP inlet forward EPC HP inlet used only as healed transfer and support of direct column interface device.

Configuration 7: Forward Pressure Regulated EPC with Inlet as Thermal Zone



Configuration 8: EPC with Auxiliary EPC Module

For more information on using auxiliary EPC see the headspace configuration in *Using valve options* later in this chapter. For further information, see *Applications of Auxiliary Electronic Pressure Control in Gas Chromatography,* HP application note 228-202, HP publication number (43) 5091 -5013E.

Using the external sampler interface with an inlet as a heated zone

The external sampler device introduces a slight pressure drop over the system. To compensate for this pressure drop, measure the actual flow with a bubble flow meter to determine the additional pressure necessary during the run. For example, measure the flow at the column outlet, or use an unretained peak to determine the average linear velocity. The pressure setting will probably be somewhat higher than would be used by the column alone.

Special considerations

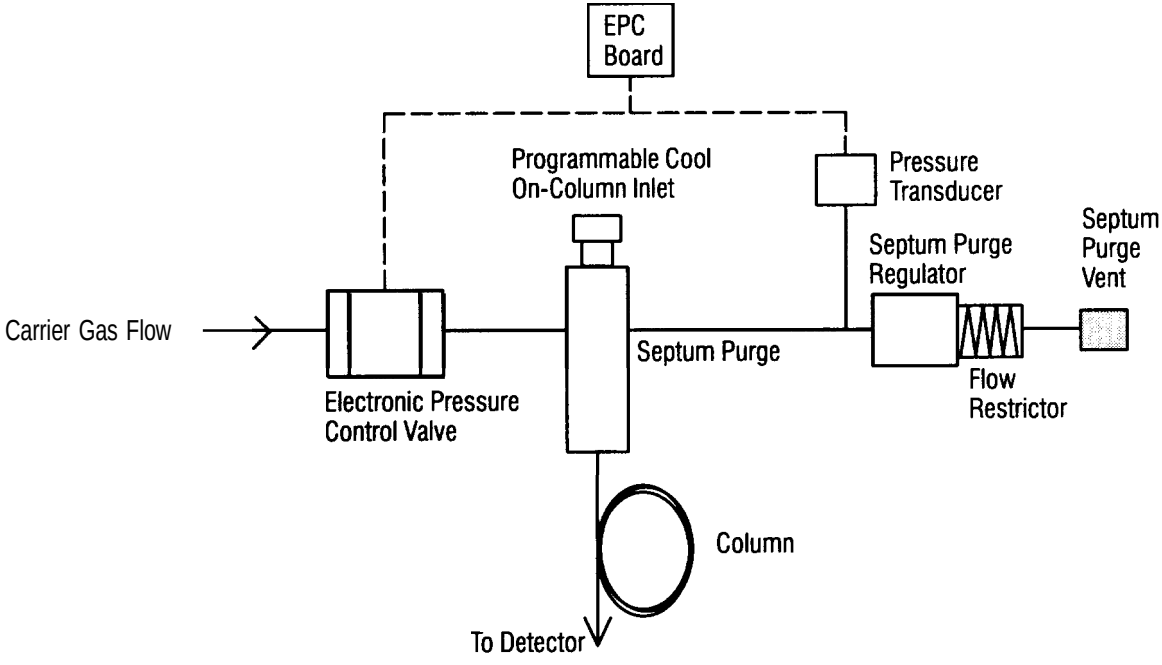
If you are using an HP 19395A Headspace Sampler with a needle interface to the inlet, replace the septum nut with the black septum nut with the wide aperture before using it.

For easier access to the external sampler interface, try to place the three-way flow diverter valve so it is reached easily through the side door of the GC.

Some devices go into a timed desorbtion cycle that increases the pressure drop through the system, consequently reducing the flow rates. To compensate for the increased pressure drop, program a pressure ramp at the beginning of the start cycle. For example, ramp the pressure from 16 psi to 35 psi at 99 psi/rein. Then program the pressure down to 16 psi for the duration of the analysis.

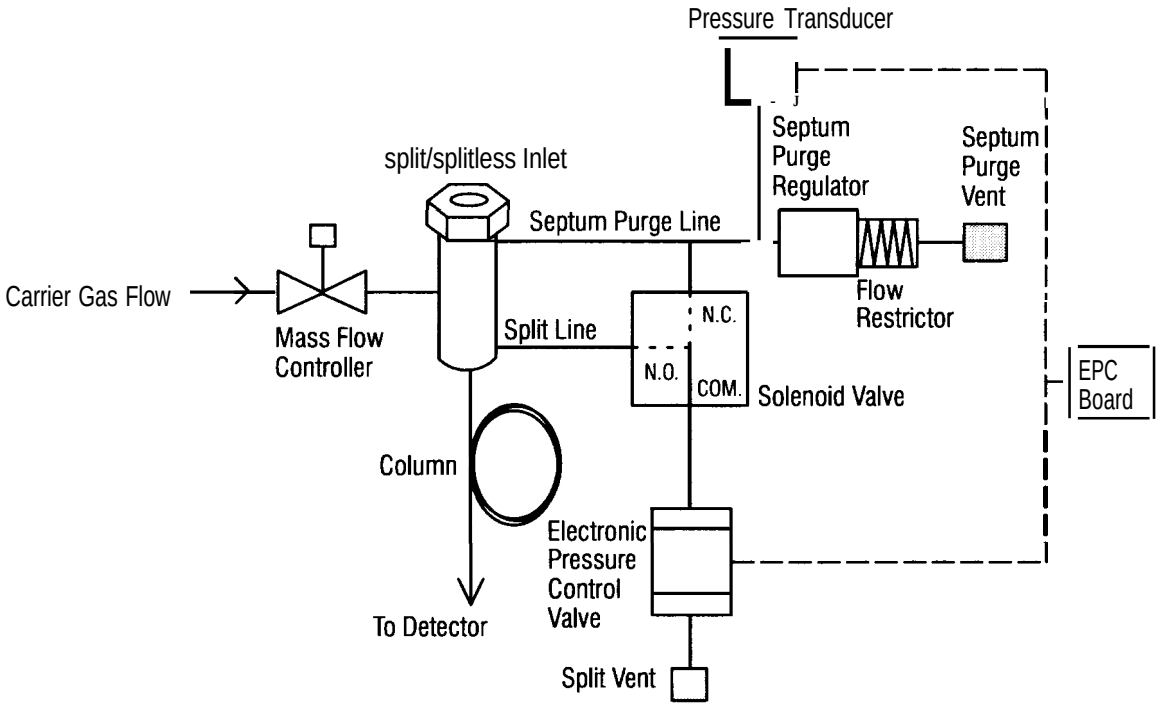
Whenever an external device is placed in series with an EPC inlet system, some of the direct flow displays will be incorrect because we do not know what the total pressure drops are relative to just the column (such as column id and length). In this case, ignore the display and measure the actual flows. If the external device switched valves, multiple columns, or traps during a run, then the flows may also change. You may want to compensate for these situations in your pressure programs.

The standard forward-pressure configuration is shown below.



Forward-Pressure Regulation for the On-Column Inlet and the Septum-Purged Packed Inlet EPC Used with Open-Tubular Columns)

The standard back-pressure configuration is shown below.



Back-Pressure Regulation for the Split/Splitless Inlet

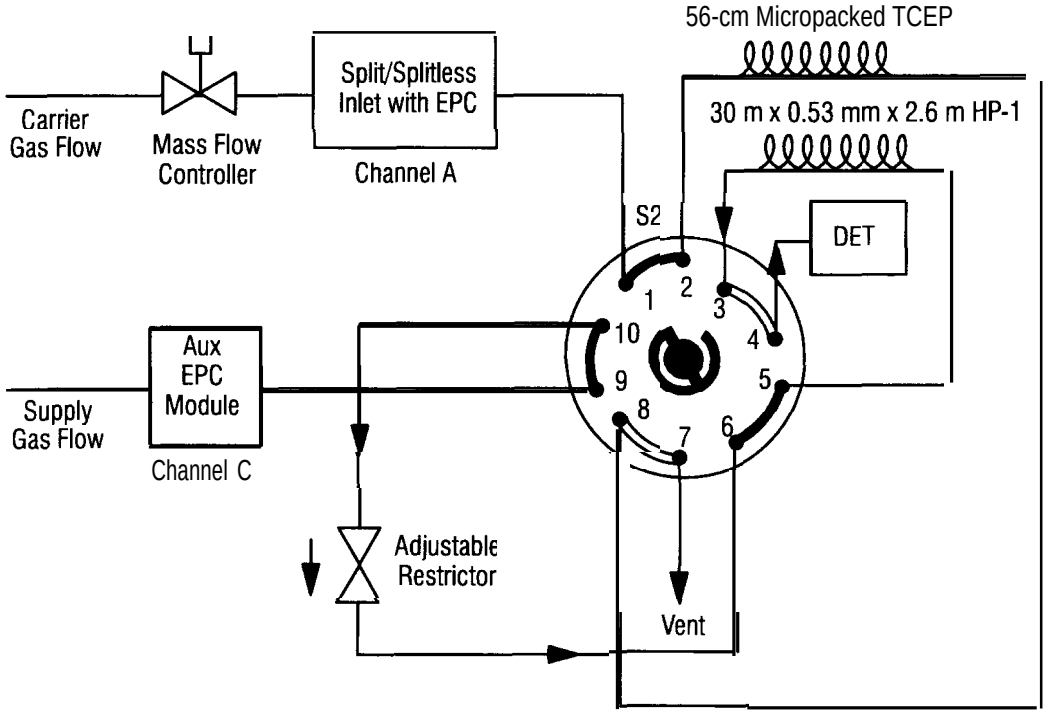
Using valve options

The valve options described in this section demonstrate several applications of the auxiliary EPC system. Hewlett-Packard supplies 17 standard plumbing configurations for the HP 5890. These configurations may be ordered through the HP 18900F Valve Ordering Guide, HP part number 5091 -4240E.

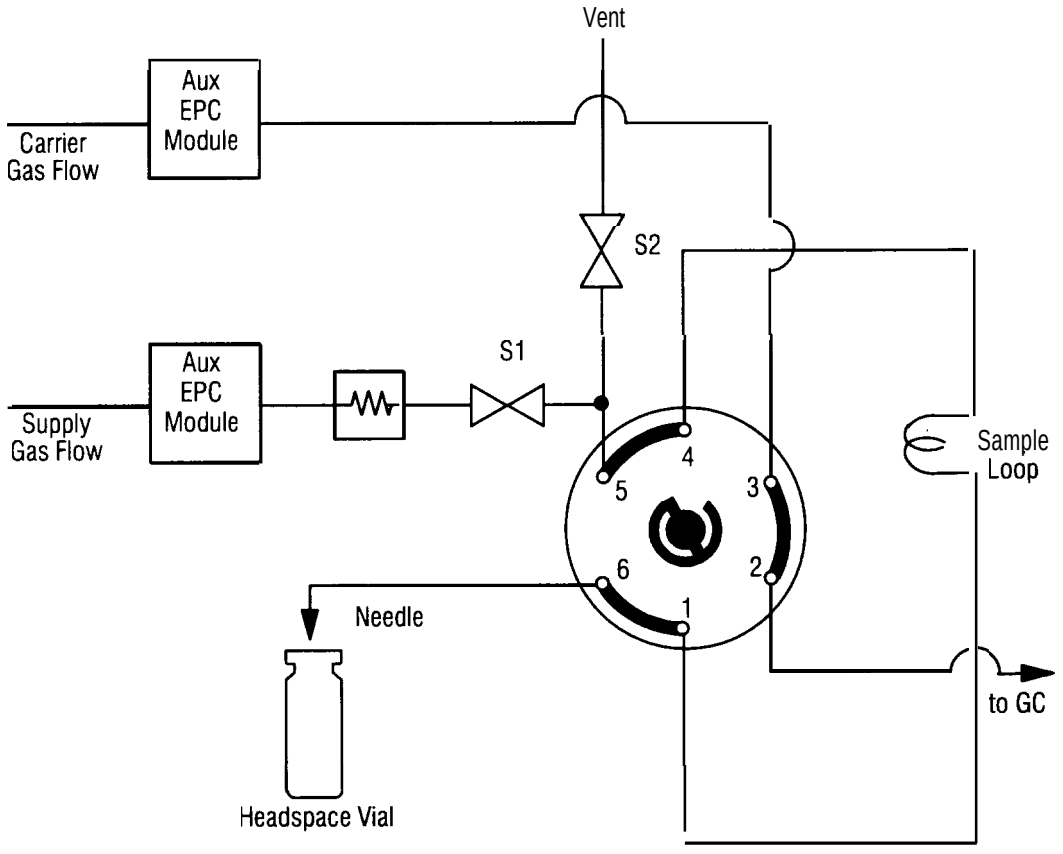
This section describes advanced auxiliary EPC valving applications that will require some method development time from the user. In general, all HP valves are compatible with auxiliary EPC However, some valve options may require additional method development time. For some configurations, such as a packed-column refinery gas analyzer that operates isothermally, an auxiliary EPC module can be used in constant pressure mode as easily as the standard mechanical pneumatics. With packed-column valve applications, be sure to check the flows with a bubble flow meter every time you change the system pressure.

If you run your system only in constant flow or constant pressure mode, without pressure programs, then an auxiliary EPC module may be just as effective as a manual flow controller for your applications. However, most operations become easier to automate once you are using EPC In fact, EPC should give faster response to changes in the back pressure of the column and better repeatability of known retention times. When constant flow mode is used with packed columns, actual flow may increase or decrease with temperature programming. It is best to calibrate the flow at the initial and final oven temperatures, and then use a two-point calibration with pressure programming.

The following diagram shows a common 10-port valve configuration plumbed with an EPC split/splitless inlet and one general-purpose auxiliary EPC module. This configuration is used for the analysis of oxygenates in gasoline.



10-Port Valve Configuration with Two Channels of EPC



Headspace Sampling Systems Product

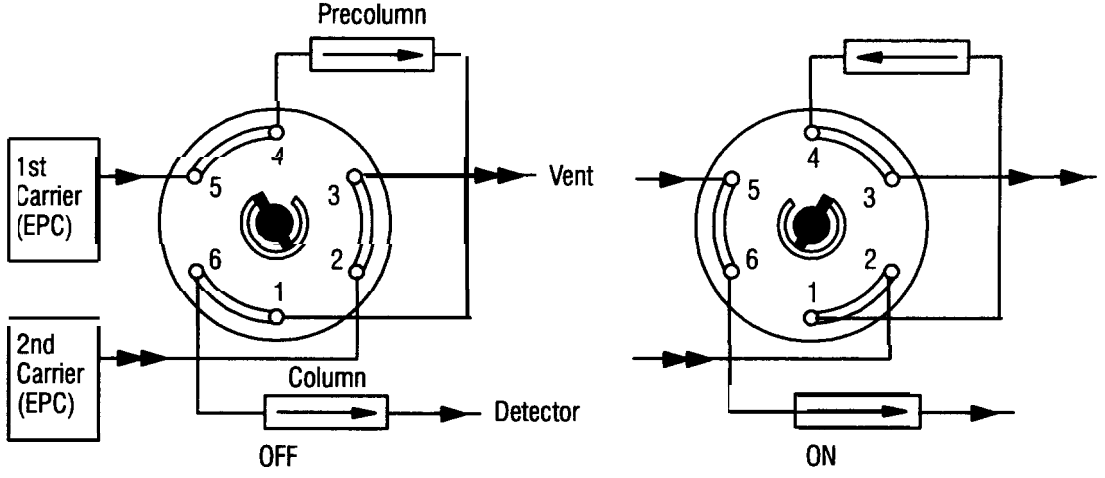
EPC can be applied to headspace sampling. The six-port valve configuration in the figure above shows the application of two general-purpose auxiliary EPC modules. One module is used for carrier gas flow, and the other module is used for precise vial pressurization.

Which valves work best with auxiliary electronic pressure control?

Almost all HP valves are compatible with auxiliary EPC and many of the standard configurations offer additional system benefits. For example, using valve options for specific applications can help you maintain better reproducibility, reduce ambient temperature sensitivity, and rapidly change the flow rate. Using valves with auxiliary EPC also contributes to easier setup and simplified automation. In addition, auxiliary EPC valves make it easier to change run times and to reduce analysis time.

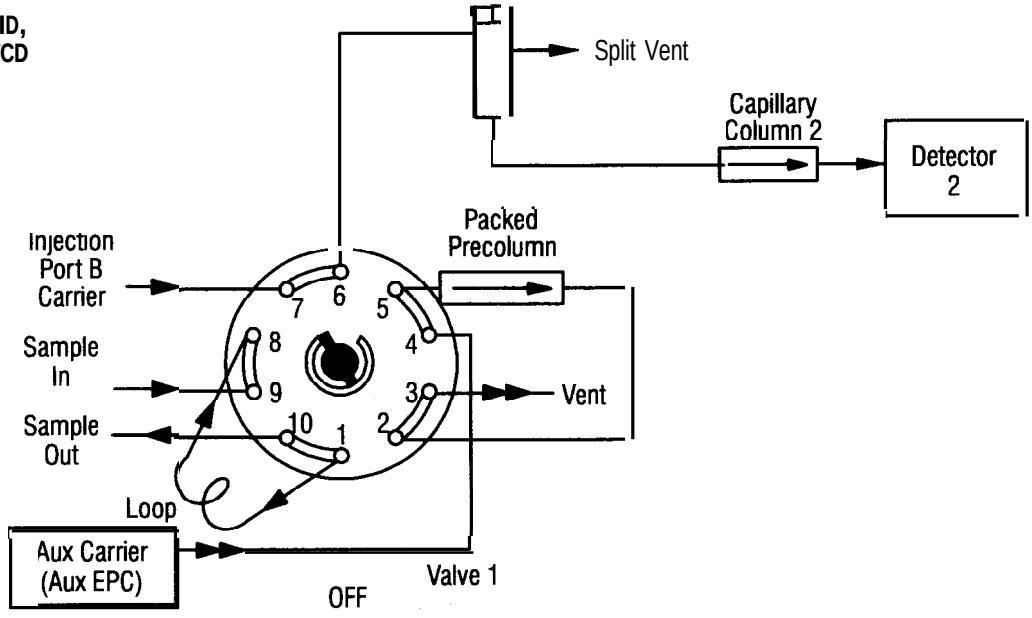
Valve configurations such as Options 205 and 401 can also be used with EPC If pressure programming is used, you may need to change the pressure program whenever you change the valve timing. Failing to change the pressure program will produce incorrect results.

Options 205 and 235 backflush precolumn



A common application has Option 401 interfaced to a capillary Split inlet. The system uses an EPC split/splitless inlet (main carrier) and one general-purpose auxiliary EPC module for control of the second carrier source. The interface between the capillary inlet and the valve is Option 901.

Option 401.
Detector 1—FID,
Detector 2—TCD



split/splitless EPC Injection Port B

Appendix A

Appendix A

Pressure versus flow relationships for inlet and auxiliary electronic pressure control

While pressure is the parameter controlled and measured with the EPC system, the corresponding column outlet flow rate and average linear velocity are also calculated and displayed on the keypad. Entries can also be made in terms of either flow or velocity, from which the system calculates the required pressure and enters this setpoint

It is important to understand how pressure, flow, and linear velocity are related so that the displayed values can be compared with each other and with the correct experimental measurements. This section includes a summary of some of the basic equations for flow through open tubular columns and the calculations that are carried out by the HP 5890 Series II GC and HP 3365 ChemStation systems. Some of the terms and units used throughout this section are shown below.

Terms and Units Used in This Section	
F	Column outlet flow, ml/min
$\bar{v}$	Average linear velocity, cm/sec
L	Column length, cm
r	Column inner radius, cm
t	Retention time, seconds
T	Column (oven) temperature, K (°C + 273)
η	Carrier gas viscosity at temperature T, poise
P_i	Inlet pressure, absolute
P_0	Outlet pressure, absolute; zero if vacuum compensation specified
T_{ref}	Reference temperature; 25°C = 298 K
P_{ref}	Reference pressure; 1 atm = 14.7 psi = 1.01325x 10^6dynes/cm^2
T_{ref} / T	2981323 = 0.923
Column	25 m x 0.32 mm, Helium carrier

Outlet flow

Column outlet flow can be calculated from Equation 1:

$$F = \frac{60\pi r^4}{16\eta L} \left[\frac{T_{ref}}{T}\right] \left[\frac{P_i^2 - P_o^2}{P_{ref}}\right] \qquad \text{Eq 1}$$

Since the gas volume depends on both temperature and pressure, it is expressed here under reference conditions, T_{ref} and P_{ref}. Flows displayed by the 5890 are based on reference conditions of 25 °C and 1 atmosphere pressure, for comparison with experimental values measured under ambient conditions with a flow meter at the outlet of the detector.

Average linear velocity

Flow cannot always be measured directly at the detector outlet, as in work with a mass spectrometer or at very low flow rates. An alternative is to measure the elution time for an unretained peak and calculate the average linear velocity for gas flowing through the column. This value is determined at oven temperature T.

$$\bar{v} = \frac{L}{t} \qquad \text{Eq 2}$$

The average linear velocity can also be calculated from pressure, temperature, and column parameters according to Equation 3, as was done for outlet flow using Equation 1. (Temperature does not appear separately, but is included in the viscosity term in this equation.)

$$\bar{v} = \frac{3r^2}{32\eta L} \frac{(P_i^2 - P_o^2)^2}{(P_i^3 - P_o^3)} \qquad \text{Eq 3}$$

This is the calculation used for the velocity value displayed by the HP 5890. It is calculated at oven temperature, corresponding with experimental measurements from elution time of an unretained peak (equation 2).

Calculating flow from average linear velocity

Combining equations 1 and 3 gives an equation that can be used to calculate flow from average linear velocity.

$$F = 60\pi r^2 \left[\frac{T_{ref}}{T}\right]\left[\frac{2}{3P_{ref}} \quad \frac{(P_i^3 - P_o^3)}{(P_i^2 - P_o^2)}\right] \bar{v} \qquad \text{Eq 4}$$

Terms for both temperature and pressure appear in this equation. The ratio T_{ref}/T corrects for the difference in temperature (ambient versus oven) at which flow and velocity were calculated. The pressure term is related to effects of the gradient from inlet to outlet pressure. Because the carrier gas is compressible, linear velocity varies along the length of the column, depending on the pressure at each point. Retention time measurements reflect the average linear velocity $\bar{v}$, but the pressure-gradient term must be included to calculate flow at the outlet.

Column flow is often approximated from measurements of unretained peak time according to equation 5, without including these corrections (see Chapter 4, *Setting Inlet System Flow Rates* earlier in this manual).

$$F \cong 60\pi r^2 \bar{v} \qquad \text{Eq 5}$$

This can be a good approximation, when the pressure drop is small and temperature is near T_{ref}. The compressibility correction becomes increasingly important as the pressure drop increases, however, and flow values approximated using equation 5 may differ significantly from those calculated by the system according to equation 4.

The examples below show how the two flow calculations compare for one column under different sets of operating conditions. In these examples, temperature has been held constant to illustrate the effect of changes in the compressibility factor. It can be seen from the equations, however, that changes in the temperature ratio will also affect the overall result and comparison between calculations.

Keyboard Displays

Example	Inlet Pressure	Flow (ml/min)	Velocity (cm/see)
1	**4.6 psig (19.3 psia)**	**1.00**	19.3
2	8.3 psig (23.0 psia)	2.00	34.5
3	14.3 psig (29.0 psia)	4.00	58.4

Example I—Inlet pressure 4.6 psig

1. Calculating flow from the average linear velocity using equation 4:

$$F = 60\,(3.14)\,(0.016)^2 \quad (0.923)\left[\frac{2}{3\,(14.7)} \quad \frac{(19.3)^3 - (14.7)^3}{(19.3)^2 - (14.7)^2}\right] \bar{v}$$

$$F = (0.0482)\,(0.923)\,[1.164]\,(19.3) = 1.00$$

2. Calculating flow from the average linear velocity using the approximation in Equation 5:

$$F \cong 60\,(3.14)\,(0.016)^2\,\bar{v}$$

$$F \cong (0.0482)\,(19.3) = 0.93$$

Example 2-Inlet pressure 8.3 psig

1. Calculating flow from the average linear velocity using Equation 4:

$$F = 60\,(3.14)\,(0.016)^2 \quad (0.923)\left[\frac{2}{3\,(14.7)} \quad \frac{(23.0)^3 - (14.7)^3}{(23.0)^2 - (14.7)^2}\right] \bar{v}$$

$$F = (0.0482)\,(0.923)\,[13.03]\,(34.5) = 2.00$$

2. Calculating flow from the average linear velocity using the approximation in Equation 5:

$$F \cong 60\,(3.14)\,(0.016)^2\,\bar{v}$$

$$F \cong (0.0482)\,(34.5) = 1.66$$

Example 3—Inlet pressure 14.3 psig

1. Calculating flow from the average linear velocity using equation 4:

$$F = 60\,(3.14)\,(0.016)^2 \quad (0.923)\left[\frac{2}{3\,(14.7)} \quad \frac{(29.0)^3 - (14.7)^3}{(29.0)^2 - (14.7)^2}\right] \bar{v}$$

$$F = (0.0482)\,(0.923)\,[1.540]\,(58.4) = 4.00$$

2, Calculating flow from the average linear velocity using the approximation in equation 5:

$$F \cong 60\,(3.14)\,(0.0\,16)^2\,\bar{v}$$

$$F \cong (0.0482)\,(58.4) = 2.82$$

References

More detailed discussions on pressure versus flow relationships in capillary GC can be found in many general references; two are listed here.

1. WE. Harris and H.W. Habgood, *Programmed Temperature Gas Chromatography,* Wiley, New York, 1966.

2. J.C. Giddings, *Unified Separation Science,* Wiley New York, 1991.

Index

D

E

F

G

H

I

L

M

N

O

P

R

S

T

V

W

Z