Reference Manual

HP 5890 Series II and HP 5890 Series II Plus

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Warranty

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 border of their premises.

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

Sound pressure Lp < 70db(A)

During normal operation

At the operator position

According to ISO 7779 (Type Test)

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

Schallemission

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

Diese Information steht im Zusammenhang mit den Anforderungen der 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

Contents

1

Columns and Fittings

Columns and Fittings

The HP 5890 SERIES II (hereafter referred to as HP 5890) provides 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.

This section provides information for the following:

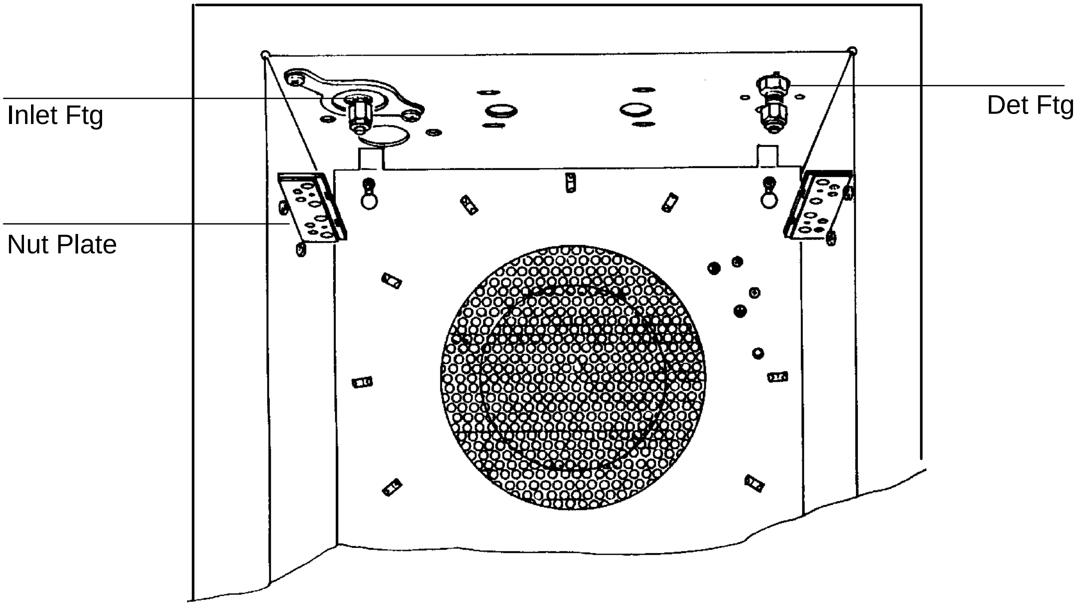
- The column oven.
- Fittings.
- Liners and inserts.
- ECD and TCD capillary makeup gas adapters.

The first three items must be considered **before** a column may be installed properly at either an inlet or a detector. In addition, for an FID or NPD, and depending upon the column to be installed (packed versus capillary), the correct jet must be installed **before** installing the column. Jet installation is described in Chapter 8, *Preventive Maintenance*.

For specific information on ordering fittings, liners, and inserts, see Hewlett•Packard's analytical supplies catalog.

Column oven

Figure 1-1



The Column Oven

The oven door latch, located beneath the lower right corner of the door, is pressed upward to open the door.

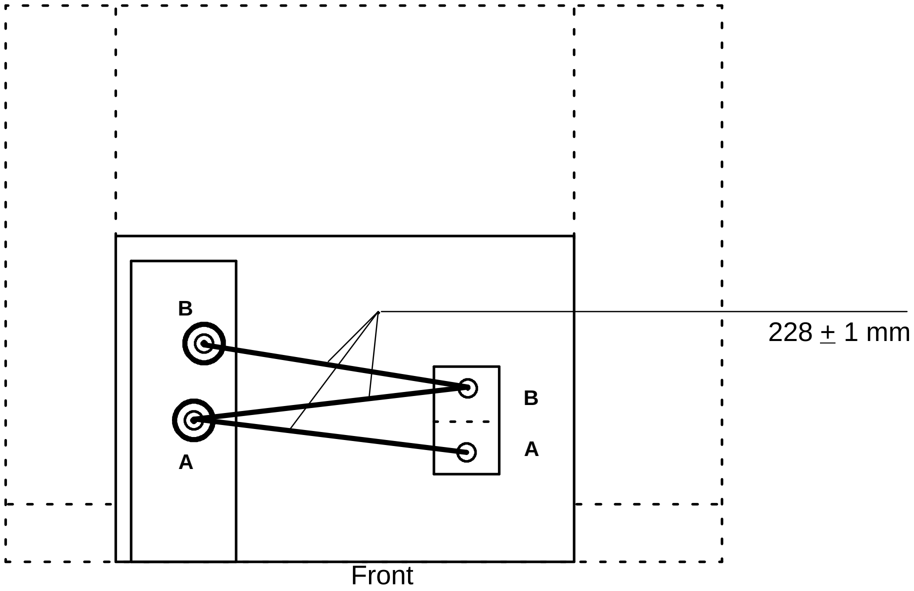
Motor•drivenflaps at the rear of the oven admit room air for cool down or near•ambientoperation, so the door is kept closed except for access to columns (the oven cools most efficiently with its door closed).

The oven can maintain temperature down to about 7°C above ambient without auxiliary cooling. If lower temperatures are required, a cryogenic valve (for either liquid CO_2 or liquid N_2) is needed. Liquid CO_2 permits reliable temperature control down to -50 °C; liquid N_2 provides reliable control down to -80 °C. The maximum oven temperature is 450°C.

Column placement

Generally, a column may be installed between any inlet and detector. A rigid 1/4•inchpacked glass column, however, if installed in the **B** (rear•most)inlet, can **only** be installed in the **B** (rear•most)detector. Distance relationships among inlets and detectors are shown in Figure 1•2.

Figure 1-2



Top View
(showing relationship of inlets to detectors)

Installation Restrictions, Rigid Columns

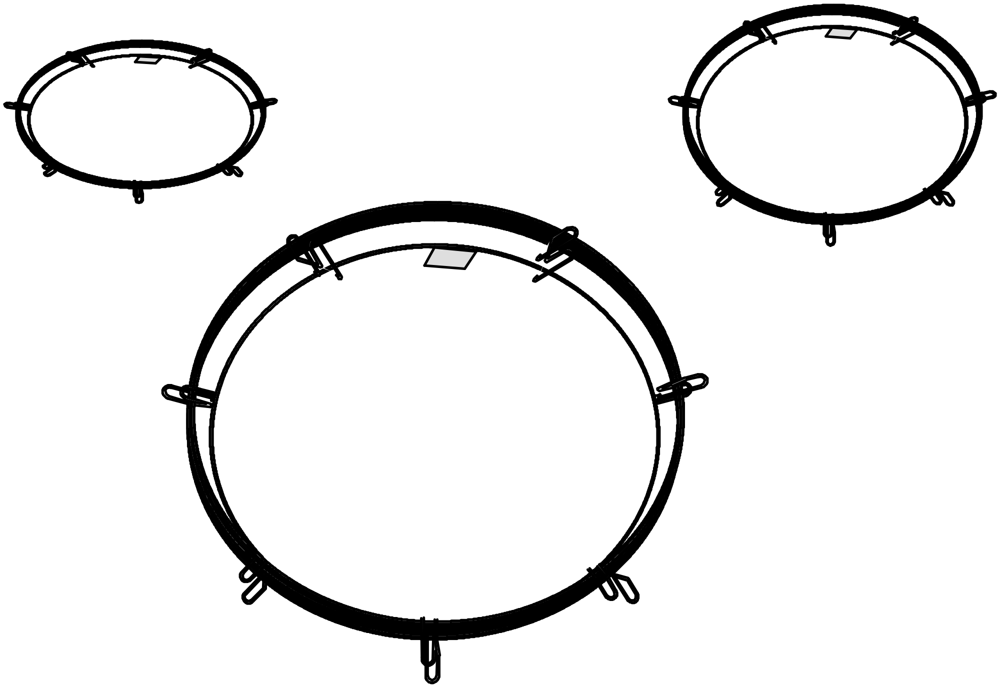
Packed column

Packed columns require no physical support other than that provided by proper installation at inlet and detector fittings.

Hewlett-Packard capillary columns

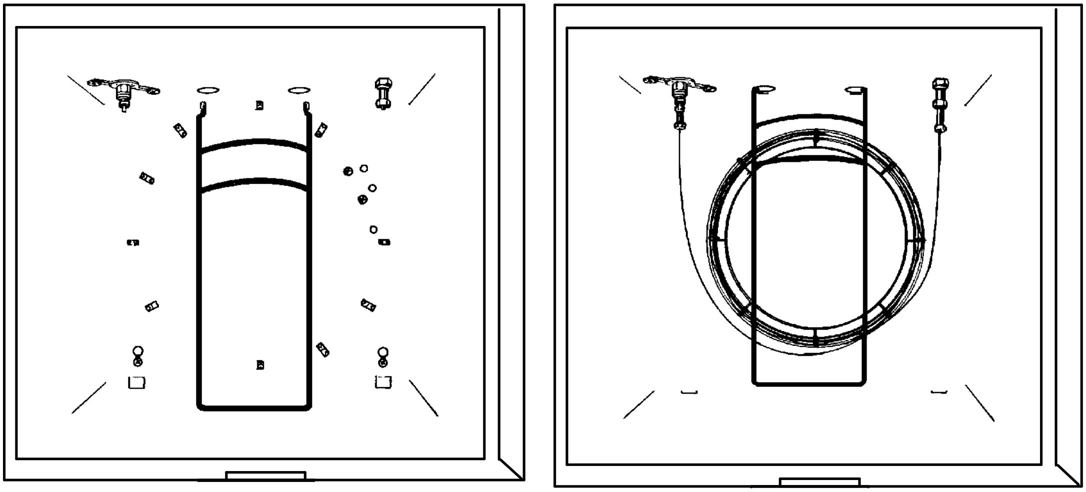
Hewlett•Packardcapillary columns are wound on wire frames which mount on a pair of brackets which slip into slots at the top of the oven interior.

Figure 1-3



Typical Hewlett-Packard Capillary Columns

Figure 1-4.



Column Hanger
Part No. 1460-1914

Column Installed

Installed Bracket for Hewlett-Packard Capillary Columns

The bracket has two positions from which to hang the column wire frame. Depending upon frame diameter, use the position which 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.

Fittings

The following is a brief comparison of common types of fittings (nuts, ferrules, O•rings)used to install columns, and to install inlet and detector liners and/or inserts. Each type has its own set of advantages and disadvantages:

- Graphite O•ringsor ferrules have excellent sealing quality and long service life, can be used continuously to 400°C, and are generally recommended for most applications, particularly capillary and glass columns. They are also recommended for inlet and detector liners, and for split/splitless capillary inlet inserts.

  Since they do not adhere permanently to glass or metal, they can be removed easily without damage to the column, tubing, liner, or insert. A rear metal ferrule may be needed if recommended by the manufacturer.

- Commonly used with metal columns and tubing, brass nuts and ferrules on the column prevent damage to inlet and detector liners, but may develop leaks above 250°C or with temperature programming.
- Also commonly used with metal columns and tubing, stainless steel fittings minimize possibility of leakage at high temperature, but require care in installing columns; overtightening may damage the column end or inlet/detector fitting.
- Teflon ferrules may be used to 250°C but are recommended only for isothermal work, because they develop leaks when temperature•programmed.
- Commonly used with glass columns, Vespel (or graphite•filledVespel) ferrules are reusable and work well to 350°C. These ferrules may leak or crack if tightened when cold.
- Glass columns can be installed using silicone O•rings. For O•ring installation, a back metal ferrule, reversed, is necessary to provide a flat surface to seal against.

  Silicone O•ringsare useful to about 250°C, but, due to bleed, interfere in high•sensitivitywork. They also gradually lose elasticity and crack, so they must be replaced fairly often.

Table 1-1. Typical Fittings for Columns and Inlet/Detector Liners, Adapters, and Inserts

Type	Description	Typical Use	Part No.
1/4-inch swage, stainless steel, pkg, 20 of each	nut front ferrule back ferrule	1/4-inch packed metal columns	5080-8753
1/8-inch swage, stainless steel, pkg, 20 of each	nut front ferrule back ferrule	1/8-inch packed metal columns	5080-8751
1/4-inch swage, brass, pkg, 20 of each	nut front ferrule back ferrule	1/4-inch packed metal columns	5080-8752
1/8-inch swage, brass, pkg, 20 of each	nut front ferrule back ferrule	1/8-inch packed metal columns	5080-8750
Vespel, pkg of 10	1/4-inch ferrule	inlet/detector liners, 1/4-inch glass packed columns	5080-8774
Vespel, pkg of 10	1/8-inch ferrule	metal columns	0100-1107
graphite, pkg of 10	1.0-mm ferrule	capillary columns	5080-8773
graphite, pkg of 10	0.5-mm ferrule	capillary columns	5080-8853
graphite	6.35-mm O-ring	inlet/detector liners, 1/4-inch glass packed columns, split capillary inlet insert	0905-0767
graphite	6.52-mm O-ring	splitless capillary inlet insert (use)	0905-1004
silicone	6.0-mm O-ring	inlet/detector liners 1/4-inch glass packed columns, split/splitless capillary inlet inserts	0905-0322
silicone	1.0 mm O-ring	capillary columns	0905-0759

Note: Dimensions given are id’s of the O-ring or ferrule.

Liners/adapters and inserts, general

A liner/adapter is installed from **below**, inside the oven; it serves both as an adapter to mate the particular column to the inlet or detector and to provide correct internal volume for proper operation.

Inserts are used with inlets only, and, when required, are installed from above, at the top of the inlet; these are discussed specifically later in this section (see *Inlet inserts*).

In general, the analysis to be performed determines the column to be used. The column then dictates hardware required for the inlet and detector (liner, insert, adapter, jet (FID or NPD)).

Note:

- A correctly designed 1/4•inchpacked glass column requires no liners since the column ends themselves serve this purpose.
- The appropriate liner/adapter, and insert if required, must be installed prior to installing a column.

Tables 1•2and 1•3summarize hardware required for various combinations of inlets, columns, and detectors.

Table 1-2. Hardware and Recommended Fittings for Packed Column Installation

	Packed Columns		
	1/8-inch Metal	**1/4-inch Metal**	**1/4-inch Glass**
Recommended Column Fittings	1/8-inch swage-type nut and ferrules[3]	1/4-inch swage-type nut and ferrules[3]	1/4-inch swage-type nut and graphic ferrule or silicone O-ring(s)
Packed Column Inlet Liners	19243-80510[1] or 19243-80530[1] (requires glass insert)	19243-80520[1] or 19243-80540[1] (requires glass insert)	None
FID/NPD[2] Liners/Adapters	19231-80521[1]	19231-80530[1]	None
TCD Liners/Adapters	None	19302-80020[1]	19302-80020[1] (may require altering the column)
ECD Liner/Adapters	19301-80530[1]	None	None

---

[1] Use 1/4-inch swage-type nut and Vespel or graphite ferrule to install liner/adapter.

[2] See Chapter 8 for details regarding jet exchange (if necessary).

[3] See information later in this chapter regarding proper installation of swage-type fittings on packed metal columns.

Table 1-3. Hardware and Recommended Fittings for Capillary Column Installation

	Capillary Columns			
	HP Series 530 µ	**320 µm ID**	**200 µm ID**	**Metal/ Glass**
Recommended Column Fittings	Capillary column nut and 1.0-mm graphite ferrule, or silicone O-ring(s)	Capillary column nut and 0.5-mm graphite or silicone O-ring(s)	Same as 320 µm	Same as **HP Series 530 µ**
Packed Column Inlet Liners	19244-80540[1] (requires glass insert)	Not Recommended	Not Recommended	Not Recommended
Split/Splitless & Split-Only Capillary Inlet Split Sampling	18740-60840 with graphite or silicone O-ring	Same	Same	Same
Split/Splitless Capillary Inlet Insert: Splitless Sampling	18740-80220 with graphite or silicone O-ring	Same	Same	Same
Programmable On-Column Capillary Inlet Insert	19245-20580[3]	19245-20520	19245-20510	19245-20550
FID/NPD[2] Liners/Adapters	19244-80550[1]	Same	Same	Same
TCD Liners/Adapters	18740-20950 and 18740-20960[3]	19232-80550[1]	Same	Same
ECD Liners/Adapters	19244-80550[1,3]	19233-80530	Same	Same

---

[1] Use 1/4-inch swage-type nut (if a nut is not supplied as part of the adapter) and graphite or Vespel ferrule to install liner/adapter.

[2] 0.11-inch jet must be used; see Chapter 8 for information regarding jet exchange (if necessary).

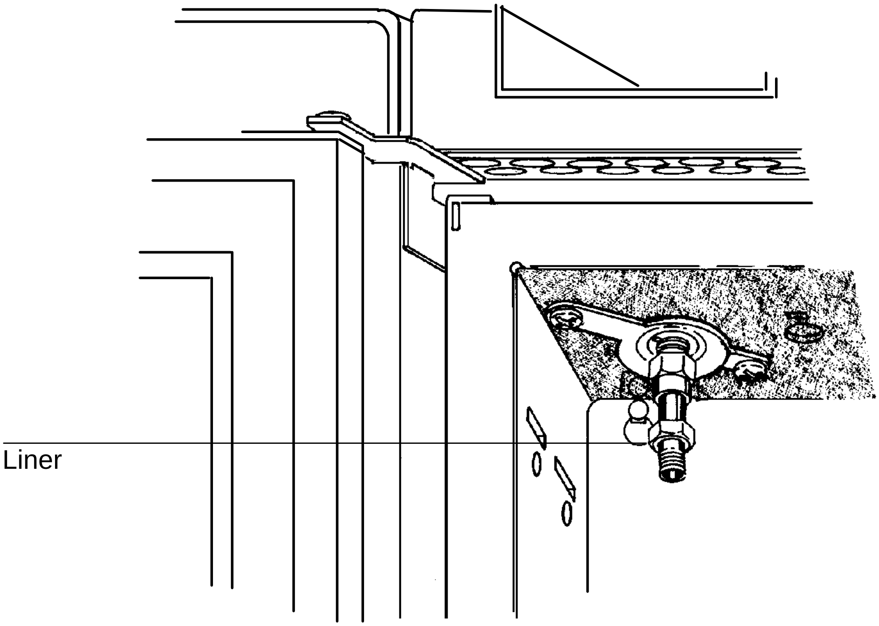
[3] Use only if detector is **not** configured with capillary makeup gas adapter. If makeup adapter is provided, it is used instead (usually with makeup gas turned off).

Inlet/detector liners/adapters

Interchangeable stainless steel liners/adapters, installed from inside the oven, are used with the packed column inlet, and with all detectors, depending upon the column to be installed.

Packed column inlet liners

Figure 1-5



Installed Liner, Packed Column Inlet

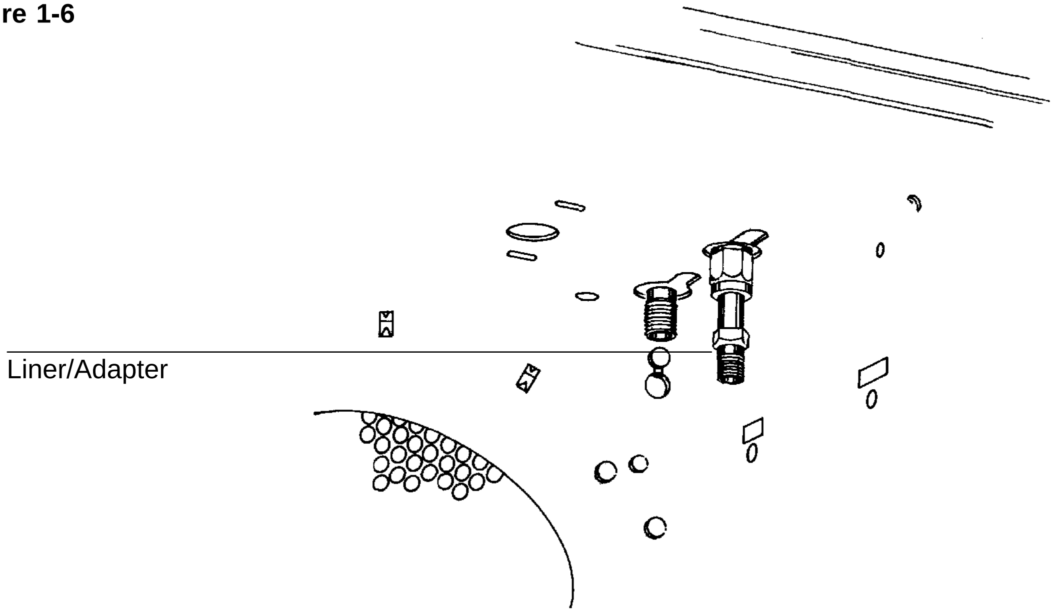
Liners for the packed column inlet are available in three sizes: one for 1/8•inchcolumns, one for 1/4•inchcolumns, and one for HP Series 530 µ capillary columns.

In addition, liners for the packed column inlet are available to accept glass inserts (discussed later) for reduced reactivity, to trap nonvolatile residues, or for use with an HP Series 530 μ capillary column.

- No liner is used with 1/4•inchpacked glass columns. The long leg of the column fits into the inlet body, replacing the liner. Packing and glass wool plug must be below the tip of the needle for best results.
- Metal columns are installed with a liner appropriate for the column diameter.
- If necessary, glass columns can be installed using a metal liner (preferably those accepting a glass insert), but this is not recommended. There may be problems with dead volume in connections, and preventing contact of sample with metal surfaces.

Detector liners/adapters

Figure 1-6



Typical Installed Detector Liner/Adapter

Detectors require a liner/adapter to be installed when used with packed metal columns (either 1/8•or 1/4•inch),and with any type of capillary column. Normally, no liner is required with 1/4•inchpacked glass columns, since the leg of the column itself serves as the liner.

For the FID or NPD, the correct detector jet must be installed prior to installation of the liner. (If jets must be exchanged, see Chapter 8, *Preventive Maintenance.*)

ECD and TCD adapters

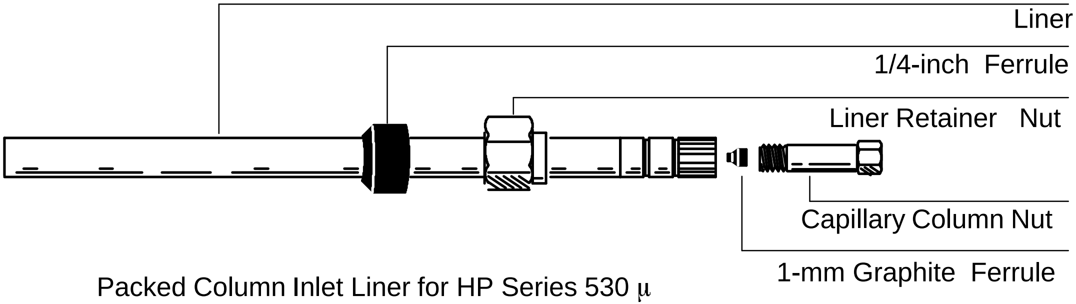
A makeup gas adapter must be installed in the ECD or TCD base to install a capillary column, and to augment carrier flow through the column with additional gas flow needed for optimal detector operation. The adapter must be removed for packed column applications.

In addition, to install an **HP Series 530**μ capillary column in an ECD or TCD having no capillary makeup gas adapter, the following adapters are used: Part No. 19244•80550 for the ECD, and Part No. 18740•20950 and 18740•20960 for the TCD.

Finally, to use a 1/4•inch column with the TCD (having a base designed for 1/8•inch columns), a 1/8•to 1/4•inch adapter is required (Part No. 19302•80020). For the ECD (having a base designed for 1/4•inch columns), to use a 1/8•inch column, a 1/4•to 1/8•inch adapter is required (Part No. 19301•80530).

Liner/adapter installation

Figure 1-7



Packed Column Inlet Liner for HP Series 530 µ Capillary Column Use

Nut and Ferrule Installed on a Liner/Adapter

With one exception, liners/adapters are installed in the same manner; if the liner/adapter has not been used before, a **new** ferrule must be installed.

The single exception is the adapter to install an HP Series 530 µ capillary column in a TCD without provision for capillary makeup gas (Part No. 18740•20950 and 18740•20960). In this case, no ferrule is required to form a seal with the detector base.

Note: A **graphite** ferrule is strongly recommended; since metal ferrules tend to **lock** permanently onto the liner/adapter, their use may require replacing the entire liner/adapter, should a permanent leak develop.

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

Note: The liner/adapter must be kept as clean as possible to prevent introducing contamination into the inlet or detector. Use a clean, lint•free cloth to remove fingerprints, etc., from the end of the liner/adapter to be inserted into the inlet or detector base. CH_3OH (methanol) may be used as a solvent.

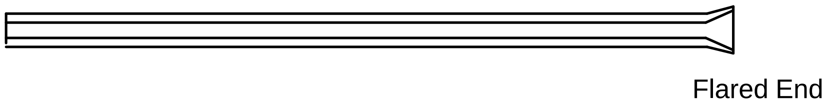
1. Assemble a brass nut and graphite ferrule onto the liner/adapter.
2. Insert the liner/adapter straight into the detector base as far as possible.
3. Holding the liner/adapter in this position, tighten the nut finger•tight.
4. Use a wrench to tighten the nut an additional 1/4 turn.
5. Install the column; then heat the oven, inlet, and detector to desired operating temperatures and, only if necessary to stop leaks, tighten fittings further.

Inlet inserts

Inserts are used in inlets, and can be installed from the top of the particular inlet.

Packed column inlet inserts

Figure 1-8

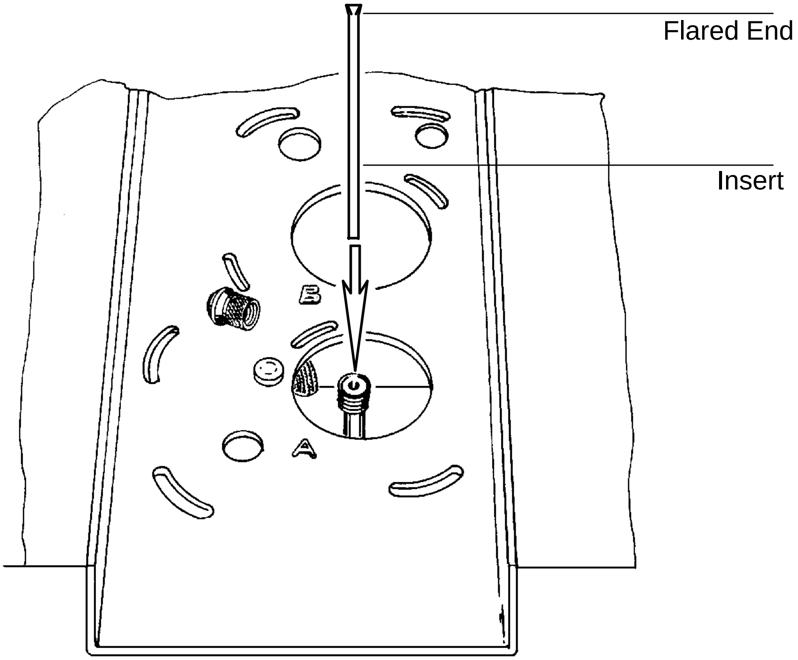


Glass Insert for Packed Column Inlet Liner

Assuming the correct inlet liner is installed, a glass insert is installed as described on the next page.

WARNING Exercise care! the oven, and/or inlet, or detector fittings may be hot enough to cause burns.

Figure 1-9



Installing a Glass Insert in a Packed Column Inlet

1. In handling the insert, avoid contaminating its surface (particularly its interior).
2. Remove the septum retainer nut and septum.
3. Carefully remove the old insert (if present) by withdrawing it straight up. A match stick or similar fibrous item may be used as an aid in lifting the insert from the inlet.
4. Install the new insert by dropping it carefully, straight into the inlet liner, flared end up.

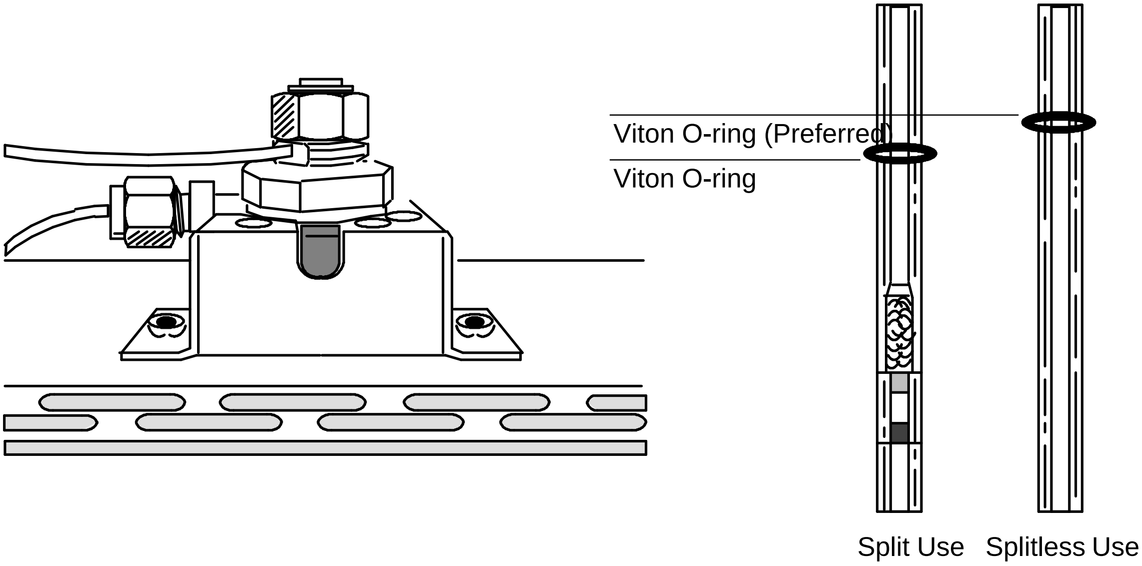
Note: For the liner and insert for an HP Series 530 µ capillary column, if the column is already installed, a new insert may not seat properly in the liner; the column may prevent it from dropping completely into the liner.

If the insert does not drop completely into the liner, **do not** force it (either the liner or the column may shatter); instead, remove the column, seat the insert, and then replace the column.

5. Replace the septum and septum retainer nut.

Split/splitless or split-only capillary inlet inserts

Figure 1-10



Split/Splitless and Split-Only Capillary Inlet and Inserts

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

- Split, for major•componentanalyses
- Purged splitless, for trace•componentanalyses

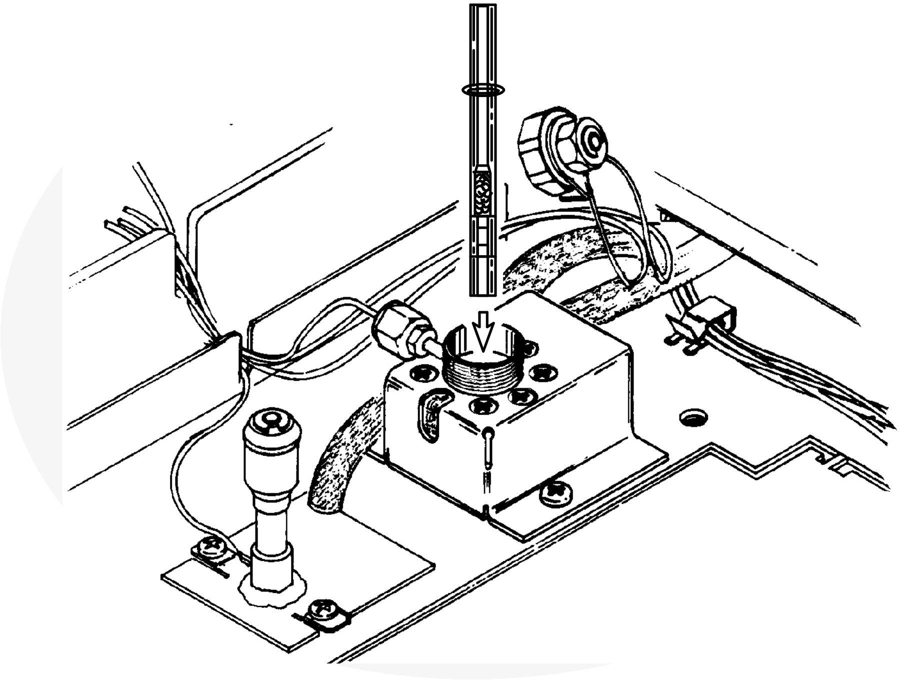
The split insert contains packing material (10% OV•1on 80/100 High Performance Chromosorb•W),held in place by silanized glass wool plugs, located immediately above a mixing chamber. This ensures proper volatilization and homogeneous mixing of the sample prior to its entry into the column.

WARNING Exercise care! The oven, and/or inlet, or detector fittings may be 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 backpressure 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.

Figure 1-11



Installation, Split/Splitless Capillary Inlet Insert

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•ringon the insert, about 2 to 3 mm from its top end.
6. Install the insert, pressing it straight down, as far as possible, into the inlet.

Caution Do not add any seal either at the bottom of the inlet or at the bottom of the insert; to do so will damage the inlet and/or shatter the insert.

7. Replace the insert retainer nut, tightening it to **firm** finger•tightness to form a leak•freeseal. Do not overtighten.

Jet replacement, FIDs or NPDs

Depending upon the column type (packed versus capillary) to be used, and/or analyses to be performed, exchanging the jet in an FID or NPD may be necessary. This must be done **prior** to column installation, and is particularly important in optimizing FID performance.

Exchanging the jet in either an FID or an NPD is described in Chapter 8, *Preventive Maintenance*.

Metal capillary columns

Most metal capillary columns (0.6 to 1.0 mm od) can be connected directly. Some metal capillaries have a large•diametersleeve soldered on each end; this must be removed. Use a small triangular file to score the tubing behind the sleeve; then bend the sleeve back and forth until it breaks.

It is important 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.

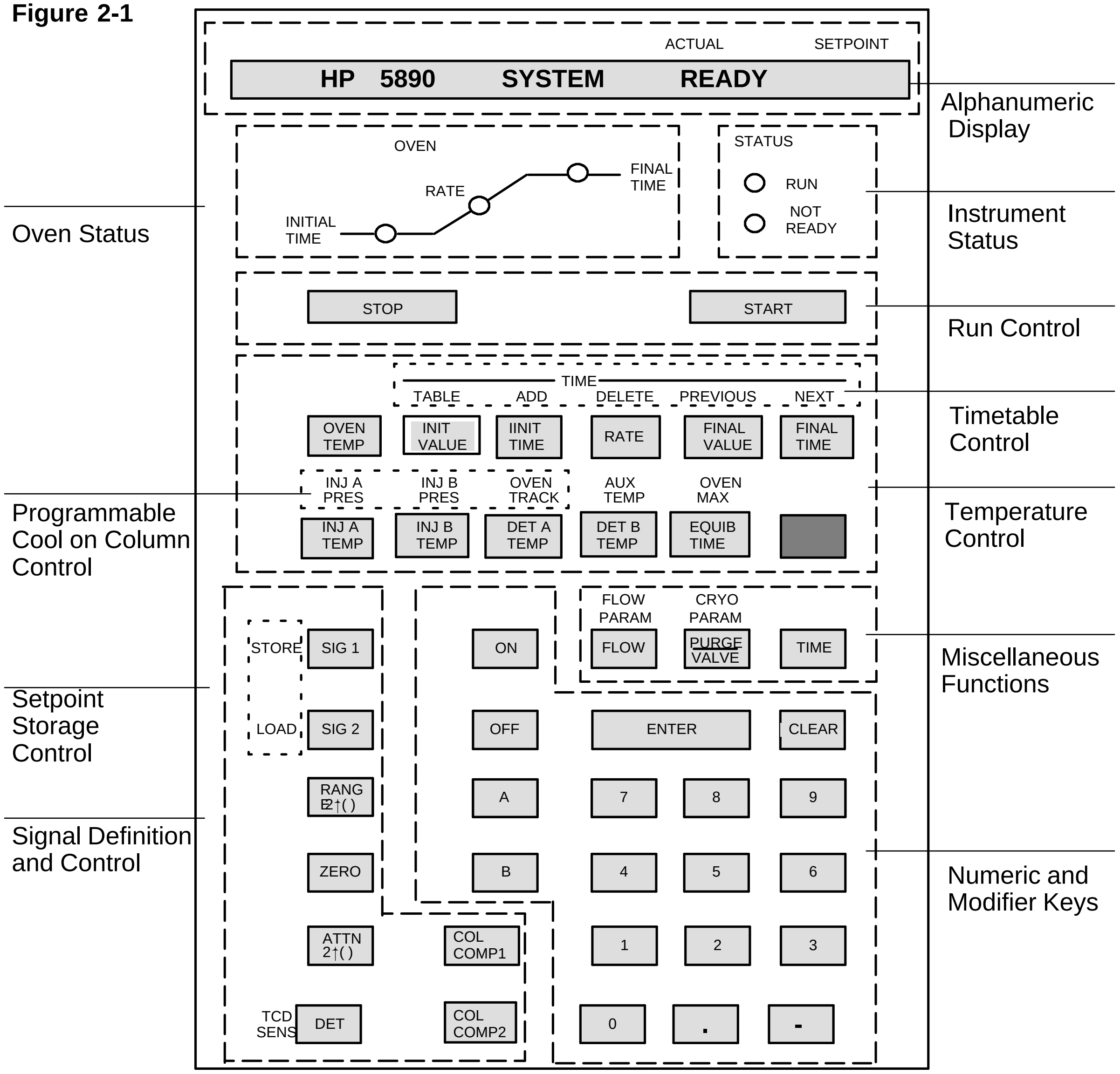
Therefore, whenever the column must be cut to provide fresh ends, use a suitable file to first score the column at the point at which it is to be broken. This is done normally **after** installing on the column the column nut and ferrule (or O•ring)required for installation.

2

Keyboard and Displays

Keyboard and Displays

Figure 2-1



HP 5890 SERIES II Keyboard and Display Panel

HP 5890 SERIES II (hereafter referred to as HP 5890) operation is monitored and controlled through its front panel keyboard, and alphanumeric and LED displays.

Some instrument functions are monitored continuously: signal levels, temperatures, carrier gas flow rates (if electronic flow sensing is installed), and inlet purge valve status (if a split/splitless capillary inlet is installed).

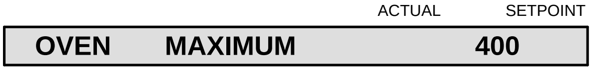
There are two general display areas:

- **Alphanumeric Display.** Echoes keys pressed at the HP 5890 keyboard show current setpoint values for instrument functions; actual values of continuously monitored instrument functions; and warning, error, information, and diagnostic messages.
- **LED Display.** Consists of two parts: the left half (OVEN) displays oven status during a run; the right half (STATUS) gives overall instrument status at any given time.

Displaying setpoints

Any particular instrument current value and/or setpoint is displayed at the alphanumeric display simply by pressing the appropriate instrument function key followed possibly by additional modifier" keys necessary to further define the function (i.e., A or B, SIG 1 or SIG 2, ON or OFF, TIME). For example, pressing OVEN MAX might give the display:

Figure 2-2



Example, a Typical Alphanumeric Setpoint Display

The name of the function key pressed is always displayed, along with the current setpoint and/or measured values.

Examples of possible displays are provided where appropriate throughout the manual.

If a particular function is **not** installed in your instrument, an appropriate message is displayed when the key corresponding to the function is pressed. For example, if no heated zone controlled by DET B TEMP is installed, pressing DET B TEMP gives the display:

Figure 2-3

ACTUAL SETPOINT

DET B NOT INSTALLED

Typical Display, a Function NOT Installed in the Instrument

Entering setpoints

To enter a setpoint value for a particular instrument function, the function is first displayed by pressing the appropriate key(s).

Once the chosen HP 5890 function is displayed, a new setpoint value can be entered at any time by pressing appropriate keys 0 through 9, ▪, -, or possibly A, B, ON, or OFF. For a numeric value, ENTER is pressed to terminate the entry. Figure 2•4summarizes the two steps involved.

To display the function and its setpoint:

Figure 2-4

(Instrument Function Key) A o B)

necessary for a few instrument functions

then, EITHER

(0 through 9 - . ENTER

to enter a new setpoint value

OR, for a few functions,

(ON or OFF)

to switch the function on or off

Steps in Entering a Setpoint Value

For example, to set the **A** detector zone to 250°C, the following sequence of keys is pressed:

DET A TEMP 2 5 0 ENTER

function key numeric key(s)

Once detector A temperature is displayed by pressing DET A TEMP, the new setpoint value may be entered at any time thereafter.

Note that the display shows a flashing * (asterisk) while the new setpoint is entered, disappearing when ENTER is pressed.

When ENTER is pressed, the setpoint value is verified and, if satisfactory, becomes the new setpoint for the function. If the entered value is not satisfactory (out of range, or inconsistent with other, related setpoints), an appropriate message is displayed. A different value may be entered immediately, without again pressing the particular function key.

CLEAR can be used anytime during an entry, prior to pressing ENTER, to erase the entry in progress. The * disappears, and the original setpoint display is restored.

Rules regarding keyboard usage are summarized below:

- An instrument function key, when pressed, is shown in the display along with its current setpoint value, and actual value for continuously monitored functions: signal levels, temperatures, flow rates.
    - A displayed function is also ready for entering a new setpoint value simply by pressing appropriate keys.
- An * (asterisk) is flashed as the new value is entered, indicating an entry in progress.
- ENTER must terminate a numeric entry. It is also required in terminating a sequence to assign a particular detector to a given output signal channel.

  In general, any display showing the flashing * must be terminated using ENTER.

  Upon pressing ENTER, the value is verified to ensure it is within the range permitted for the particular function, and/or that the value is internally consistent with previously defined setpoint values for other, related functions.
    - If the value is accepted, * disappears from the display indicating the new value is stored and implemented. For functions whose values are continuously monitored, * is replaced by the actual value.
    - If the value is out of range, or inconsistent with another setpoint value, an appropriate message is displayed. Another setpoint value may be entered immediately without again pressing the particular instrument function key.

  The original setpoint value remains in force until an acceptable entry is made.

- CLEAR is used anytime during setpoint entry, prior to pressing ENTER, to erase the entry in progress.
- CLEAR, if pressed when **no** setpoint entry is in progress, displays HP 5890 readiness .
- Run Control Key START, if pressed while a setpoint entry is in progress, causes the entry to be aborted.
- If a particular key is not valid, it is simply ignored if pressed during setpoint entry.
- While an entry is in progress, other instrument function keys are ignored if pressed, until the current entry is terminated and stored (ENTER) or erased (CLEAR).

Keyboard operation, INET control

In general terms, HP 5890 operation is the same whether the instrument is under local control or INET control (controlled by a separate device). If the HP 5890 is to be controlled through INET, the following should be noted:

- In the event communication is lost (e.g., by power lost at one or more devices on the loop, a disconnected INET cable, etc.), HP 5890 START and STOP keys will be disabled.
- Should the HP 5890 keyboard exhibit problems (keys inactive) while under INET control, disconnect INET cables at their HP 5890 receptacles; then switch power to the HP 5890 off, **and** then on. The keyboard should behave normally (assuming the HP 5890 itself has no problems).
- To restore INET control, check that all devices on the loop are powered on, and that all INET cables are installed properly. The system should return to normal operation automatically.

Additional information regarding INET control is available in Chapter 5, *Signal Output***.** Servicing may be required for one or more devices on the INET loop if communication cannot be established.

Protecting setpoints

The HP 5890 provides a keyboard **lock** feature to minimize possibility of stored setpoints being altered unintentionally. When the HP 5890 keyboard is **locked**, setpoint values (numeric values, A, B, OFF, and ON) may **only** be displayed; they **cannot** be altered. START and STOP remain functional, so runs may be started or stopped.

To **lock** instrument setpoints, first enter the key sequence:

CLEAR ■ - ENTER

Then press ON to lock the keyboard, or OFF to unlock the keyboard. Figure 2•5shows displays occurring during this process.

Figure 2-5

```
                         ACTUAL     SETPOINT
CALIB      AND       TEST        [0-0]

                         ACTUAL     SETPOINT
KEYBOARD      LOCK      ON

                         ACTUAL     SETPOINT
KEYBOARD      LOCK      OFF
```

Keyboard Lock Displays

With the keyboard locked, Figure 2-6 shows the display occurring if a setpoint entry is attempted:

Figure 2-6

ACTUAL SETPOINT

KEYBOARD LOCKED

KEYBOARD LOCKED Message Display

If the HP 5890 keyboard is **locked** while the instrument is under INET control, a setpoint file may be loaded into HP 5890 memory from the controller, but the loaded setpoints cannot then be edited at the HP 5890 keyboard until it is unlocked.

After locking or unlocking the keyboard, return to operation by pressing any function key (e.g., OVEN TEMP).

Loading default setpoints

This function permits resetting HP 5890 operating setpoints to a standard set of values. **Only** the following information is retained:

- Calibration constants for oven temperature control
- All gas flow rate monitoring information (gas types and calibration constants)
- Column compensation data, including detector assignments

Note: Since user-defined setpoints are lost in the process, any critical HP 5890 setpoints should be recorded for later reentry before proceeding.

Through the keyboard, select CALIB AND TEST mode, function 6:

CLEAR ▪ 6

Upon pressing ENTER , default setpoints are loaded into memory, erasing setpoints already present. Table 2•1lists resulting HP 5890 default setpoints.

Table 2-1. HP 5890 Default Setpoints

Function	Default Setpoint
Inj Temp (A & B):	50°C, OFF
Det Temp (A & B):	50°C, OFF
Oven Temp:	50°C, OFF
Oven Max Temp:	400°C
Cryo Cooling:	OFF
Cryo Blast	OFF
Equib Time:	3 min
Init Temp:	50°C
Init Time:	650 min
Oven Prog Rates:	0°C/min
Final Temp:	0°C
Final Time:	0 min
Inlet Purge:	ON
Purge Time:	0 min
Detector (A & B):	OFF
Signal 1 Det:	Detector **A**
Signal 2 Det:	Detector **B** if BOTH detectors **A** and **B** are installed; otherwise, detector **A**.
Range (1 & 2):	0
Attn (1 & 2):	0, ON
Zero (1 & 2):	0, ON
HP-IL (INET):	Address- 31
INET:	**Global** (unconfigured)
Keyboard Lock:	OFF
Timetable	Empty
Inj A Pres	OFF
Inj B Pres	OFF
TCD Sens	HIGH
Oven Track	ON
Constant Flow	OFF

Note that if the battery protecting memory should fail when main power is turned off, the default setpoints are loaded into memory when the battery is replaced. In addition, calibration constants for oven temperature control and gas flow rate monitoring are also reset to default values.

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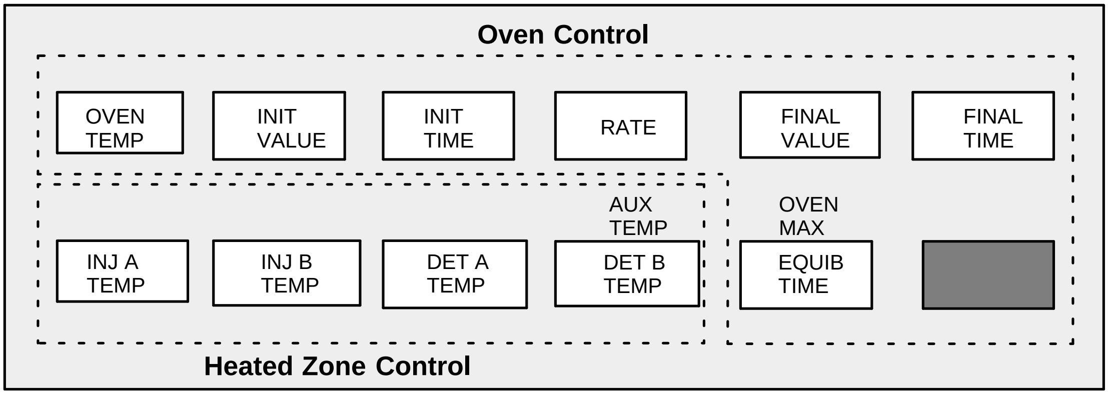
3

Temperature Control

Temperature Control

Oven temperature, and temperatures of up to five separate heated zones (detectors, inlets, and/or heated valves), are controlled through keys shown in Figure 3•1.

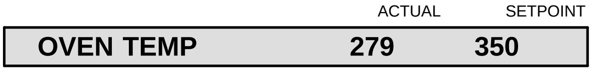
Figure 3-1



Temperature Control Keys

In these cases, **both** current setpoint value **and** current monitored value are displayed by pressing the appropriate temperature control key. For example, Figure 3•2shows typical displays obtained by pressing OVEN TEMP.

Figure 3-2



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 used in defining setpoint values, ON, OFF, A, and B are used in certain specific key sequences:

- 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.
- 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.

Valid setpoint ranges

Table 3•1lists valid setpoint ranges for the 13 keys controlling oven and heated zone temperatures.

Table 3-1. 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	0 to 400*	1°C	Zone Control
AUX TEMP	0 to 400	1°C	Zone Control

NOTE: TOTAL run time will not exceed 650.00 minutes regardless of values entere
INIT TIME , RATE , an FINAL TIME .

---

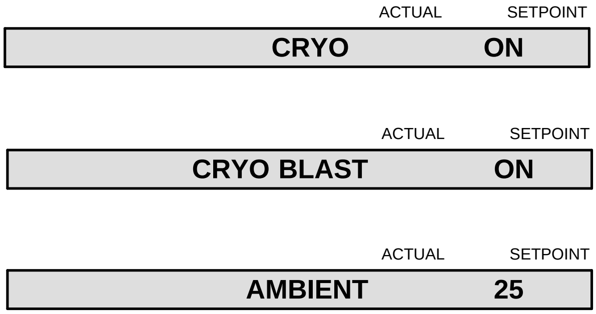
*The valid setpoint range for a Flame Ionization Detector is 0 to 450°C.

Cryogenic (sub-ambient) oven control

Liquid N_2 or liquid CO_2 cryogenic options are for operation at temperatures less than about 7°C above ambient. This is done through operation of a valve which opens when coolant is demanded and closes when the setpoint temperature is reached.

When you press gold CRYO PARAM CRYO PARAM CRYO PARAM you scroll through a series of displays for choosing cryogenic options. These options include CRYO for operation during the entire run, CRYO BLAST, for very fast cool down between runs, and AMBIENT to regulate on and off times to optimize coolant use.

Figure 3-3



For operation during runs at subambient temperatures.

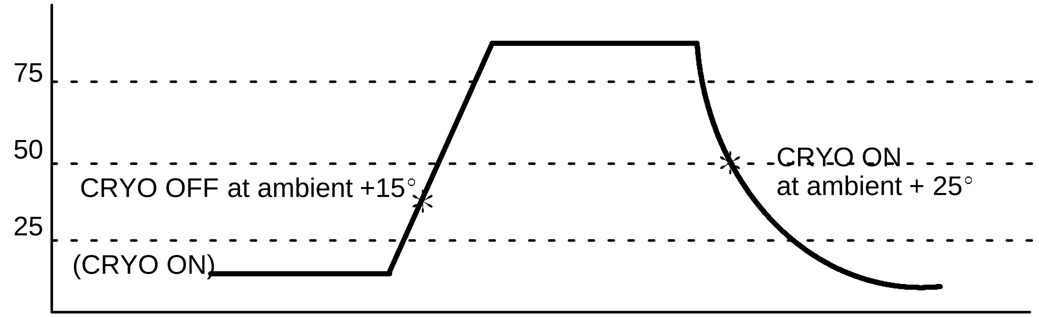
For very fast cool down between runs.

Lets you regulate Cryo and Cryo Blast on and off times to optimize coolant use. Default is 25°C and need not be changed unless
ambient differs by 10°.

Cryogenic options

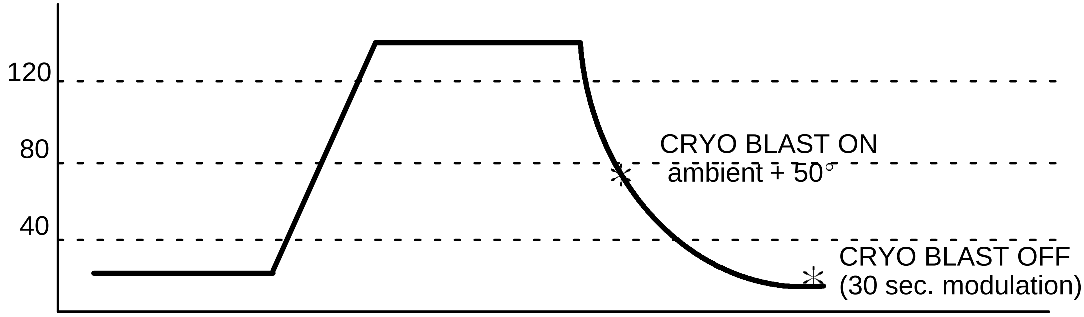
The following figures show the oven temperature profile for a typical run, showing the on and off times for CRYO and CRYO BLAST.

Figure 3-4



Oven profile using CRYO, for operation during runs at subambient temperatures

Figure 3- 5.



Oven profile using CRYO BLAST, for very fast cool down between runs

Programming oven temperature

HP 5890 oven temperature programming allows up to three ramps, in any combination of heating or cooling. Keys defining an oven temperature program include:

INIT TEMP	A setpoint temperature value at which the oven is maintained at the beginning of a temperature•programmed run. This is also the temperature to which the oven returns at termination of the temperature•programmedrun. When **not** in a run, the setpoint value for INIT TEMP equals OVEN TEMP .
INIT TIME	Time for which oven temperature is held at INIT TEMP .
RATE	Rate at which the oven is to be heated or cooled.
FINAL TEMP	Temperature the oven attains at the end of a heating or cooling ramp. In a multiple•ramptemperature program, final temperature for one ramp is also the **initial** temperature for the next ramp.
FINAL TIME	Time period over which oven temperature is held at FINAL TEMP . In a multiple•ramptemperature program, final time for one ramp is also the **initial** time for the next ramp.

total elapsed time for a run cannot exceed 650 minutes: at 650 minutes, the run terminates and oven temperature recycles to INIT TEMP . To know calculated total length of time anticipated for a run, TIME is pressed repeatedly until a NEXT RUN display is obtained.

In isothermal operation (RATE = 0), if INIT TIME is set equal to 0 (zero), the HP 5890 internally sets run time to the maximum, 650 minutes.

A is included in key sequences defining parameters for a second ramp; B is included in key sequences defining parameters for a third ramp.

In isothermal operation, and in one- or two-ramp temperature programs, rate for the **next** ramp **must** be set to 0 (zero) to prevent further programming.

In oven temperature programming, once any one of the five temperature programming functions (INIT TEMP , INIT TIME , RATE , FINAL TEMP , and FINAL TIME) is displayed, pressing ENTER , without entering a new setpoint value, **rolls** the display successively through the entire group, (including A and B displays for second and third ramps).

This is an efficient way in which to review and, if necessary, change oven temperature program setpoints.

Oven status

During a temperature-programmed run, the LED OVEN display provides indication of oven status at any given time:

- **Isothermal Run:** Assuming INIT TIME is assigned a value greater than 0, and that RATE = 0, only the INITIAL TIME LED is lit. It remains lit throughout the run.
- **Single-Ramp Temperature Program:** The three LEDs, INITIAL TIME, RATE, and FINAL TIME, successively light to indicate position in the temperature program.
- **Multiple-Ramp Temperature Program:** The three LEDs, INITIAL TIME, RATE, and FINAL TIME, successively light to indicate position in the first temperature program ramp.

  Then RATE and FINAL TIME LEDs light alternately as the program proceeds through the second (and third) ramp(s).

In complex two•or three•rampoven temperature programs, information as to the part of the program in progress is monitored by pressing OVEN TEMP.

Note that, during a ramp, the SETPOINT value displayed is that **calculated** to be the correct temperature, based upon specified heating/cooling rate, and initial and final oven temperatures.

Also, note that if the RATE LED is observed to blink during oven heating, this indicates the particular RATE value entered is too aggressive for the given operating conditions: the oven heater is operating at full power and may not be able to deliver the desired temperature program rate. Such a situation compromises accuracy in repeating the heating ramp from run to run.

Oven safety

Normally, the oven should be switched off (OVEN TEMP OFF) prior to accessing the oven interior (e.g., to change columns, check for leaks, etc.). For safety, this turns off power to the oven heater, fan, and cryogenic valve (if installed), but maintains the setpoint value in memory.

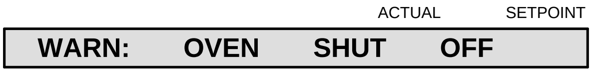
The oven is equipped with a shut•offfeature to protect against unintentional opening of the oven door, and/or the possibility of mechanical and/or electronic failure affecting oven operation.

At any time during normal above•ambientoperation, if the oven cannot attain and/or maintain an entered setpoint temperature, a problem is assumed and the oven is automatically switched off. Examples of possible problems include the oven door open (or closed but not properly latched), inoperative oven vent flaps, failure of the oven fan, heater, or temperature sensor, or electronic problem.

WARNING If the oven door is opened, a time delay may be observed before the oven shuts itself off. The closer the oven is to ambient temperature, the longer the delay will be.

The message displayed when this occurs is shown in Figure 3•6.

Figure 3-6



WARN: OVEN SHUT OFF

Message, Oven SHUT DOWN

The oven remains off until switched on again via the keyboard (OVEN TEMP ON), unless a FAULT: message is displayed (see below, *Fault: messages*). Power to the instrument must be switched off, and then on again to restore operation (setpoints are maintained).

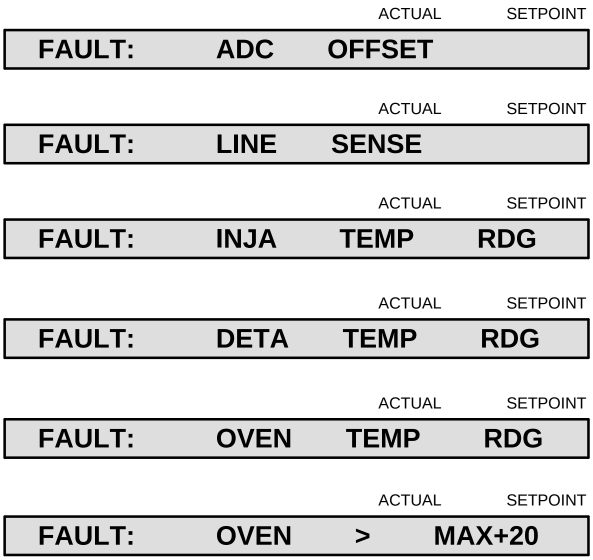
Fault: messages

Figure 3•7shows possible FAULT: messages associated with heated zones or the oven. In general, the following problems are indicated when a FAULT: message appears:

- ADC OFFSET indicates a problem with one or more electronic components in circuitry associated with temperature control.
- LINE SENSE indicates a problem with AC power to the instrument (an excessively high source voltage).
- Any of the TEMP RDG messages indicate an inoperative temperature sensor for the indicated zone or oven.
- OVEN > MAX + 20 indicates oven temperature exceeds the current OVEN MAX setpoint value by more than 20°C. Thermal run•awayis the likely cause.

Note: In case multiple problems exist simultaneously, press CLEAR to **roll** through all message displays.

Figure 3-7



FAULT: ADC OFFSET

FAULT: LINE SENSE

FAULT: INJA TEMP RDG

FAULT: DETA TEMP RDG

FAULT: OVEN TEMP RDG

FAULT: OVEN > MAX+20

Thermal Control FAULT: Messages

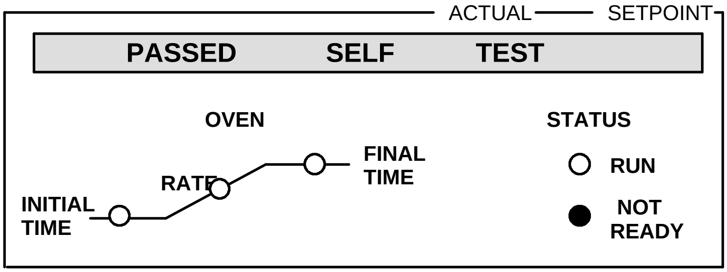
In addition to the message, the red NOT READY LED blinks. All zones and the oven are turned off and made inoperative until power is switched off, and then on again (setpoints are maintained).

After a power failure . . .

Setpoint values are protected during a power failure (even if intentional, by disconnecting the power cord, or by switching off the HP 5890 at its main power switch) by a lithium battery (10•year nominal life) which maintains power to HP 5890 memory.

After power is restored, a message is displayed, as shown in Figure 3•8.

Figure 3-8



Message Display, Power Failure and Recovery

Heated zones return to their respective setpoint values, after which the oven returns to its setpoint value.

- If OVEN TEMP is displayed after recovery from a power failure, and if the oven was ON before the power failure, the oven display shows the actual oven temperature value, and cycles between showing the setpoint value and OFF until other zones achieve their respective setpoint temperatures.
- The oven can be switched ON through the keyboard at any time, without waiting for heated zones to first come to correct temperature.
- An analytical or column compensation run in progress at the time of a power failure is aborted; similarly, a keyboard entry in progress is aborted.

Oven temperature calibration

To maximize precision with respect to retention time information, particularly if retention times are to be compared between chromatographs, it may be necessary to calibrate oven temperature in the range of interest using an independent temperature•measuring device.

With the factory•set calibration difference value of 0 (zero), displayed oven temperature is accurate to within 1% of the actual temperature, expressed in °K (Kelvin).

The HP 5890 provides the means to (if necessary) reset oven temperature monitoring so the displayed ACTUAL value accurately represents the correct temperature.

Oven temperature calibration requires entering the difference (**delta**) value (in °C) between an independently measured temperature value versus the corresponding displayed oven temperature value:

Correction Value =

Measured Temperature (°C) - Displayed Temperature (°C)

For example, if actual measured oven temperature were found to be 148.73°C, while the corresponding displayed value was 150.00, the calibration difference value to be entered would be -1.27.

Setting the oven calibration value

An oven temperature calibration measurement should be made at a temperature in the middle of the range of interest. Allow ample time (up to 1/2•hour)for thermal equilibration at the selected temperature; no drift should be observed. The temperature•sensingprobe should be placed in the region of the oven occupied by the column(s).

1. Through the keyboard, select CALIB AND TEST mode, function 1:

   CLEAR ▪ 1 ENTER

   CALIB is displayed, followed by two values: the observed oven temperature (to 0.01°C), and the current difference (**delta**) calibration value.

Note: Record the displayed calibration **delta** value! If problems are encountered in recalibration, the value may be reentered.

2. Assuming no drift in temperature, the new difference (**delta**) value is then entered by pressing appropriate number keys, followed by ENTER:

   appropriate value ENTER

3. CALIB DELTA is displayed until ENTER is pressed; then oven temperature recalibration occurs. Note that, after calibration, the displayed oven temperature value should match closely the measured value.

Any **delta** value within the range -10.00 through +10.00°C may be entered. If a value outside this range is entered, the message CORRECTION TOO HIGH is displayed.

Assuming the battery protecting HP 5890 memory is operational, a new calibration constant remains in effect even if the instrument is switched off, or disconnected from its power source, or if power fails.

4

Electronic Flow Sensing

Electronic Flow Sensing

Two channels of electronic flow rate sensing continuously monitor gas flow rates (usually carrier) in the HP 5890 SERIES II. Proper scaling of displayed values for different commonly used gases is defined through keyboard entries. The two flow channels are distinguished through A and B.

If carrier gas flows are monitored, A implies flow through column **A** (nearest the instrument front); B implies flow through column **B** (nearest the instrument rear).

Displayed flow rate values are in ml/minute.

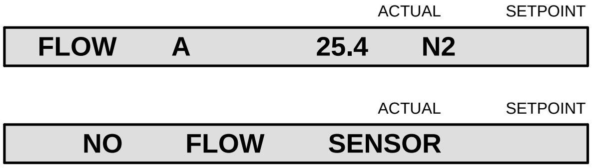
Displaying gas flow rate

Current flow rate is displayed by pressing:

FLOW A (or B)

Typical gas flow rate displays are shown in Figure 4•1:

Figure 4-1



ACTUAL SETPOINT

FLOW A 25.4 N2

ACTUAL SETPOINT

NO FLOW SENSOR

Typical Electronic Flow Rate Sensor Displays

Designating gas type

To scale the displayed flow rate value properly, one of four commonly used gases must be designated. The appropriate gas type is selected according to Table 4•1:

Table 4-1. 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

To select one of these gases for a particular flow channel, press:

FLOW (B) to display FLOW A (or FLOW B).

1, 2, 3, or 4 is then pressed, followed by ENTER. Upon pressing ENTER, the current flow rate is displayed, scaled appropriately for the chosen gas type.

If a gas **other** than one of the above **standard** four is used, select He, N_2, H_2, or Ar/CH_4 according to which one is **closest** in thermal conductivity to the gas being used. Under **no** circumstances should any corrosive gas be passed through the EFS.

The maximum usable range for H_2 is 100 ml/minute. At higher flow rates (>100 ml/min), where a gas other than He, N_2, or Ar/CH_4 is being used, or to ensure maximum accuracy in displayed flow rate, calibration of the EFS may be necessary.

Electronic flow sensor (EFS) calibration

Electronic flow sensor (EFS) calibration may be performed any time to ensure displayed flow rate accurately represents real gas flow rate through the sensor. The EFS is factory•calibratedfor four **standard** gases, H_2, He, N_2, and Ar/CH_4, within the flow rate range of 0 to 100 ml/min. This covers the majority of chromatographic applications.

Two situations where it would be appropriate to perform recalibration would be where a **nonstandard** gas is to be used (e.g., something **other** than H_2, He, N_2, or Ar/CH_4), or if flow rates in excess of 100 ml/min are to be used.

EFS calibration requires setting two values for a given flow channel•first, the **zero** value (defined with **no** flow through the given flow channel) and then the **gain** value (calculated, based upon a measured flow rate value).

WARNING If calibration is being performed for H_2, observe proper safety precautions to prevent fire or explosion hazard.

Prior to performing the calibration procedure, the following must be done:

- The instrument must be on for at least one hour for thermal equilibration of the EFS.
- Since gas flow through the channel to be calibrated will be interrupted, detectors should be turned off (particularly an NPD or TCD!), and the oven cooled to ambient temperature (to protect columns).
- A flow•measuringdevice is required, accurate to better than 1 ml/min.
- The EFS is calibrated to measure volumetric flow at standard temperature and pressure. Flows measured at ambient temperature with a bubble flow meter will have to be converted from ambient temperature and pressure to standard temperature and pressure.

Preparation

1. Access the EFS by removing the left side panel; remove two screws along its lower edge, slide the panel toward the rear of the instrument, and then lift.
2. Through the keyboard, select CALIB AND TEST mode, function 2:

   CLEAR • 2 ENTER

   GAIN A is displayed, followed by two values: the observed flow rate through Channel A, and the current **gain** calibration value for Channel A.

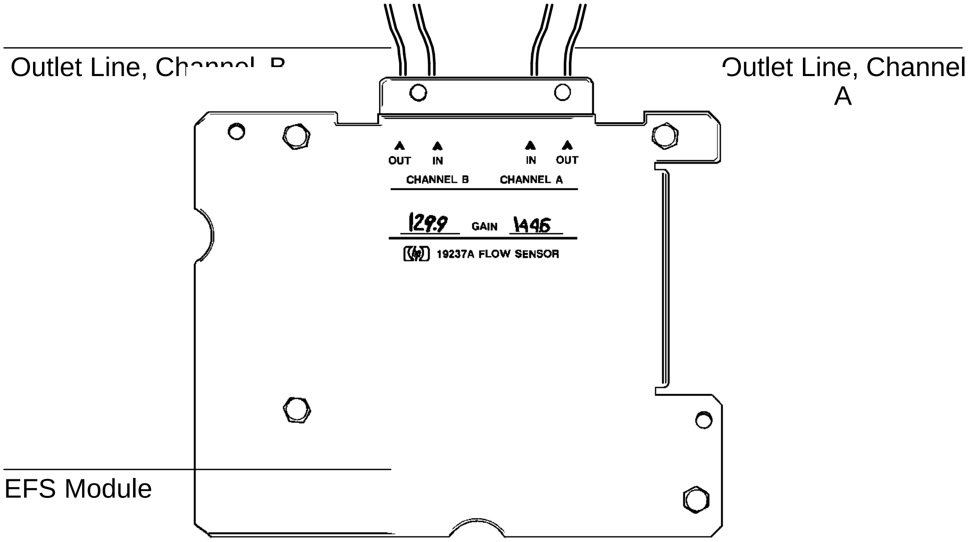
Setting the zero calibration value

The **zero** calibration value **must** be set with **no** gas flow through the channel being calibrated.

1. Press ZERO : FLOW A ZERO is displayed, followed by a value (the current **zero** calibration value for EFS Channel A). Note that Channel A is assumed by default; if channel B is to be calibrated instead, press B .
2. **Disconnect** the gas source to the particular flow channel being calibrated. **Do not** trust an on/off valve, pressure regulator, or mass flow controller to be an effective shutoff device; **any** gas flowing through the EFS will invalidate the **zero** calibration value. Disconnect the source at any convenient point (e.g., at the connection of the supply line into the instrument).

3. Locate the EFS module and note its labelling: CHANNEL A/ CHANNEL B, IN/OUT. For the channel being calibrated, locate and disconnect its OUT fitting; use two wrenches in opposition to prevent twisting the tubes.

Figure 4-2

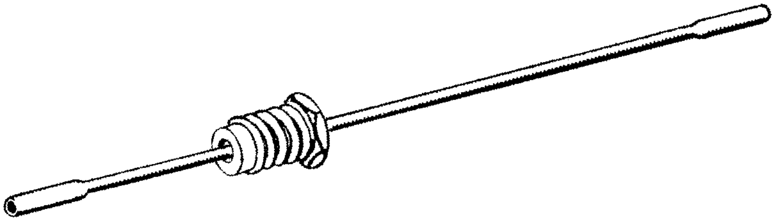


Detail, Electronic Flow Sensor (EFS) Module

4. Install the EFS flow•measuring adapter (Part No. 05890•80620) into the female OUT fitting to the EFS module. Connect a bubble flow meter to the adapter.

   Allow ample time (up to 1/2•hour) for residual gas within connected plumbing to **bleed off**. Verify that absolutely **no** flow is observed at the connected bubble flow meter.

Figure 4-3



EFS Flow-Measuring Adapter (Part No. 05890-80620)

5. Assuming there is no gas flow through the channel being calibrated, press ENTER at the keyboard. This updates the **zero** calibration value.

Setting the GAIN calibration value

After the **zero** calibration value is set at zero flow rate through the given channel, the **gain** calibration value must be set, based upon a measured flow rate.

1. At the keyboard, press FLOW : GAIN A (or GAIN B) is displayed, followed by two values (the observed flow rate through the channel, and the current **gain** calibration value for the channel).
2. Reconnect the gas supply to the channel being calibrated. **Do not** reconnect the OUT fitting for the particular channel.
3. Using a suitable flow•measuringdevice (accurate to better than 1 ml/min) connected at the OUT fitting for the given channel, adjust flow through the channel so measured flow rate is approximately in the middle of the range to be used. For example, if the range of flow rates to be used is between 50 and 150 ml/min, measured flow rate should be adjusted to about 100 ml/min.

Note: The HP 5890 has a **timer** function that may be used as an aid in measuring flow rate (see the *Operating Manual*, Chapter 4).

- Press TIME to access the **timer** function.
- After obtaining the desired flow rate, press:

  CLEAR • 2 ENTER to return to setting the **gain** value.
- EFS channel A is assumed. Press B if Channel B is being calibrated.

4. Allow ample time for flow rate to equilibrate (no drift should be observed).
5. Assuming no drift in measured flow rate, note the flow rate value at the connected flow•measuringdevice. Enter this measured value through the keyboard:

   Measured Value ENTER

   Upon pressing ENTER, CALIBRATING is displayed.
6. After a short time, GAIN A (or GAIN B) is again displayed, followed by the observed flow rate and a new **gain** calibration value based upon the measured flow rate.

   Note that the displayed flow rate value now should be quite close to the measured flow rate value. If not, drift may have occurred, so the process should be repeated.
7. This completes EFS calibration. Remove the flow•measuringadapter, reconnect channel OUT fittings (use two wrenches in opposition to avoid twisting tubes), replace the left side panel, and restore the instrument to service.

Entering specific ZERO and GAIN values

Calibration values for **zero** and **gain** should be recorded when a particular channel is calibrated. They can then be reentered through the keyboard if necessary, without repeating the entire calibration procedure.

To enter specific **zero** and **gain** calibration values:

1. Select CALIB AND TEST mode, function 2:

   CLEAR ■ 2 ENTER

   GAIN A (or GAIN B) is displayed, followed by two values (the observed flow rate through the channel and the current **gain** calibration value for the channel).

   Note that Channel A is assumed by default. If Channel B is to be calibrated instead, press B .

2. Enter the desired **gain** calibration value, preceded by - :

   - *Numeric Value* ENTER

   - is necessary to signify entry of a **gain** calibration value, rather than a measured flow rate.

3. Press ZERO : FLOW A ZERO is displayed, followed by a value (the current **zero** calibration value for EFS Channel A).

   Note that Channel A is assumed by default. If Channel B is to be calibrated instead, press B .

4. Enter the desired **zero** calibration value:

   Numeric Value ENTER

Note that alternately pressing ZERO or FLOW displays either the **zero** calibration value or the gain calibration value for the given channel (A or B).

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5

Signal Output

Signal Output

A standard signal channel, controlled via SIG 1, always is provided. A second signal channel, controlled via SIG 2, is provided if Option 550/ Accessory 19242A (**Communications Interface Board**), or Option 560/ Accessory 19254A (**RS•232**), is installed.

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.

For either channel, two separate levels of ANALOG output are provided, depending upon the analog signal cable used: +1 V or 1 mV.

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.

Zeroing signal output

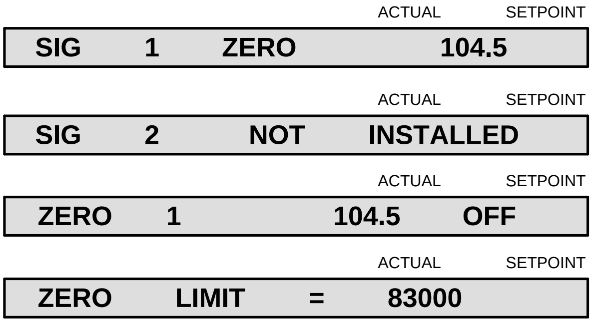
Acting on **+1 mV** and **+1 V** analog outputs, ZERO defines a constant offset which is then subtracted from the output signal (prior to signal attenuation by RANGE 2↑() or ATTN 2↑()).

The function of ZERO is to subtract 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).

Displaying current ZERO setpoint

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 in Figure 5•1.

Figure 5-1



```
                                ACTUAL      SETPOINT
SIG     1     ZERO                104.5

                                ACTUAL      SETPOINT
SIG     2       NOT      INSTALLED

                                ACTUAL      SETPOINT
ZERO     1              104.5      OFF

                                ACTUAL      SETPOINT
ZERO     LIMIT     =     83000
```

Typical ZERO Displays

Table 5•1lists conversions between detector units and the scale used for ZERO setpoint values.

Table 5-1. Conversions from Detector Units to ZERO Setpoint Values

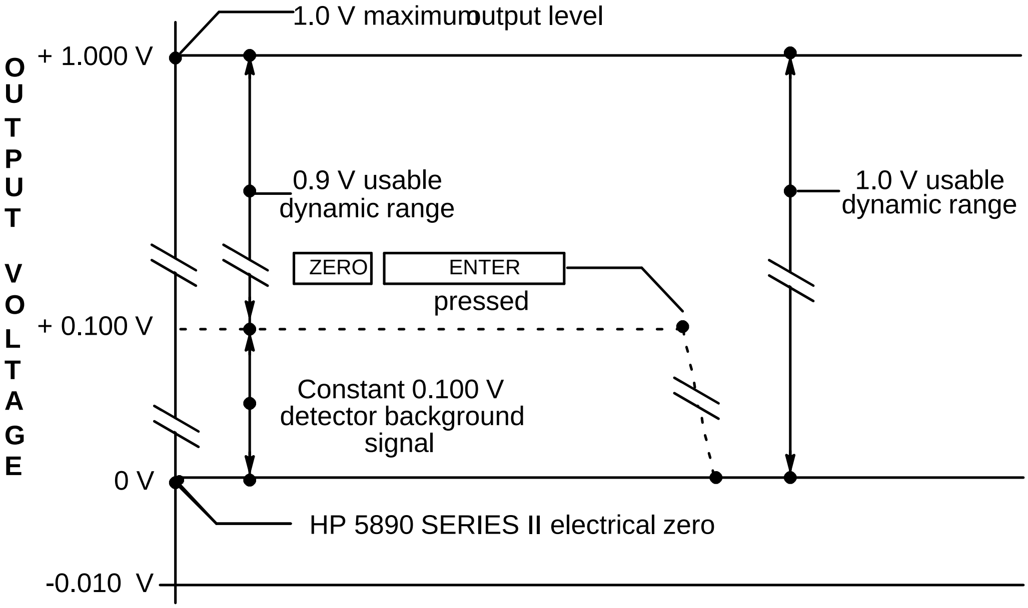
Detector	**Conversion (1.0 ZERO unit =)**
FID & NPD	1.0 pA (1.0 × 10^{-12} A)
TCD	25 µV (2.5 × 10^{-5} V)
ECD	10 Hz

Self-ZERO setpoint

Referencing Figure 5•2for the **+1 V** output, note that using ZERO can increase dynamic range available for signal output by shifting an existing constant offset signal to a lower level (usually electrical zero). There are limits to this, however, so it is good practice to have background reduced as much as possible by minimizing column bleed, using clean supply gases, and by performing proper detector maintenance.

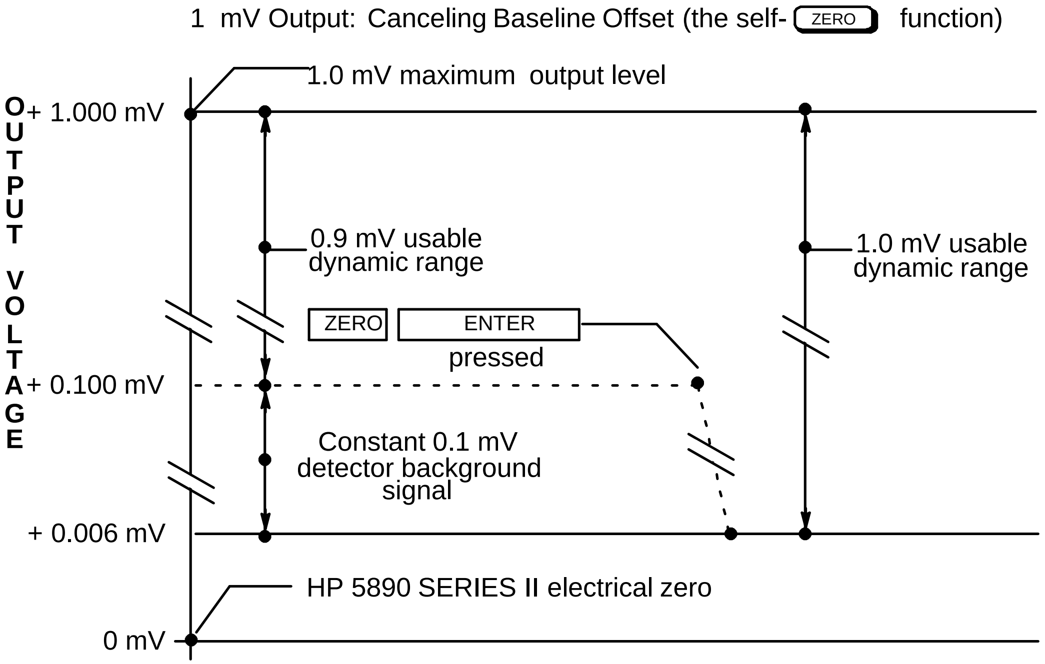
Figure 5-2

1 V Output: Canceling Baseline Offset (the s ZERO function)



Effect of ZERO on the +1 V Analog Output

Figure 5-3



Effect of ZERO on the +1 mV Analog Output

The example in Figure 5•2is in terms of the **+1 V** analog output. In Figure 5•3,identical treatment is made for the **+1 mV** output, except that the signal is shifted to an offset somewhat above electrical zero. This ensures a small positive offset (about 6% of full scale) in pen position at a connected chart recorder, and permits uninterrupted plotting, even if a small negative baseline drift occurs.

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.

Self•ZERO 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.

Note: If a self•ZERO determination is performed on an active signal exceeding the maximum permitted setpoint value for ZERO (see *User•defined* ZERO *setpoint*), the maximum setpoint value is assigned and the message SIG 1 (or 2) ZERO TOO HIGH is displayed.

User-defined ZERO setpoint

If the self•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 self•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.

Signal attenuation

Analog signal output levels (**+1 mV** or **+1 V** outputs) for either signal channel (controlled by SIG 1, and by SIG 2 if Option 550/Accessory 19242A or Option 560/Accessory 19254A is installed) are attenuated via RANGE 2↑() and ATTN 2↑().

For RANGE 2↑(), each step to a higher setpoint value decreases the output signal level by a factor of 2 (half the previous level). Also, RANGE 2↑() affects signal level at **both** the **+1 V** and **+1 mV** analog outputs.

$$\textbf{+1 V Output Level} = \frac{\textbf{Signal}}{2^{\text{RANGE } 2\uparrow()}}$$

ATTN 2↑() affects **only** the **+1 mV** analog output: each step to a higher value reduces the output signal level (as defined by RANGE 2↑()) by half.

$$\textbf{+1 mV Output Level} = \frac{\textbf{Signal}}{2^{\text{RANGE } 2\uparrow()} \times 2^{\text{ATTN } 2\uparrow()}}$$

Thus, signal output level at the **+1 mV** analog output may be set separately from that at the **+1 V** output.

Table 5•2gives values permitted for either function, and the output affected.

Table 5-2. Valid Setpoints for RANGE 2↑() a ATTN 2↑()

Key	Permitted Setpoints	Affected Output
RANGE 2↑()	0 to 13	Both +1 mV & +1 V
ATTN 2↑()	0 TO 10 OFF , ON	Only +1 mV

Note: ATTN 2↑() functions only for the **+1 mV** output, and acts on the signal **after** it has been **ranged** by RANGE 2↑()**.**

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↑() normally should 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**.

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.

For analytical information from a detector, proper settings for RANGE 2↑() and ATTN 2↑() are determined such that peaks of interest are **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. Table 5•3lists maximum detector output producing +1 volt at the **+1V** output for each RANGE 2↑() setpoint value.

Table 5-3. Detector Output Producing a 1 V Signal

	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	●	20
2	4.0×10^{3}	●	●	40
3	8.0×10^{3}	●	●	80
4	1.6×10^{4}	●	●	160
5	3.2×10^{4}	●	●	320
6	6.4×10^{4}	●	●	●
7	1.3×10^{5}	●	●	●
8	2.6×10^{5}	●	●	●
9	5.1×10^{5}	●	●	●
10	1.0×10^{6}	●	●	●
11	2.0×10^{6}	●	●	●
12	4.1×10^{6}	●	●	●
13	8.2×10^{6}	●	●	●

From Table 5•3,note that for a TCD, RANGE 2↑() = 0 is suitable for virtually all applications since 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.

Displaying current RANGE 2↑() / ATTN 2↑() setpoints

Current setpoint value for RANGE 2↑() or ATTN 2↑() is displayed by pressing the appropriate signal channel key (SIG 1 or SIG 2), followed by either RANGE 2↑() or ATTN 2↑() (or simply press RANGE 2↑() or ATTN 2↑() alone, if the desired signal channel is already displayed).

For example, the following key sequence displays current setpoint for RANGE 2↑() for the SIG 2 output channel:

SIG 2 RANGE 2↑()

Typical displays that occur are shown in Figure 5•4.

Figure 5-4

```
                                        ACTUAL       SETPOINT
 SIGNAL   1     RANGE                          10

                                        ACTUAL       SETPOINT
    SIGNAL    1      ATTN                          10

                                        ACTUAL       SETPOINT
 SIGNAL   1     ATTN      10          OFF

                                        ACTUAL       SETPOINT
 SIG  2              NOT       INSTALLED
```

Typical Signal Channel Displays

Note that if RANGE 2↑() or ATTN 2↑() is pressed without first pressing SIG 1 or SIG 2, SIGNAL 1 channel is assumed (and displayed). If desired, SIG 2 can then be pressed to display the same function for the SIGNAL 2 channel.

Entering RANGE 2↑() / ATTN 2↑() setpoints

A new setpoint value is entered for either RANGE 2↑() or ATTN 2↑() using the key sequence:

SIG 1 (or SIG 2) RANGE 2↑() (or ATTN 2↑()) *Numeric Value* ENTER

Once channel and function are displayed, appropriate keys for the new value are pressed, followed by ENTER to terminate the entry.

Switching off the +1 mV output

The **+1 mV** signal output can be switched **off**, providing no signal to the data•receivingdevice. 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

The third example display in Figure 5•4is typical after pressing 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.

Test signal output

A test chromatogram, consisting of three peaks, is permanently stored in the HP 5890. Each peak is approximately 1/10 the height of the previous peak, with the first (tallest) peak having a height value of about 125 mV at RANGE 2↑() = 0 (**+ 1 V** analog output); half•heightwidth of this peak is about 0.13 minutes.

Figure 5-5

HP 5890:

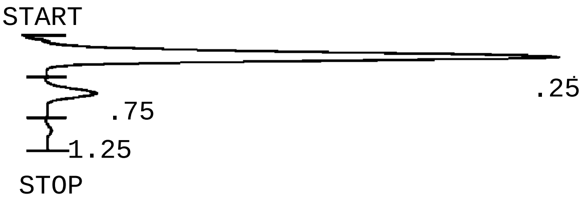
RANGE 2 ” ( ) = 0

ATTN 2 ” ( ) = 0

1 V Analog Output

```
LIST:  LIST
PEAK CAPACITY:  1159

ZERO    =  0, •1.5
ATT 2↑  =  7
CHT SP  =  1.0
PK WD   =  0.13
THRSH   =  0
AR REJ  =  0
```



```
RUN #   8


  AREA%
     RT      AREA  TYPE   AR/HT    AREA%
   0.25  79394 00    BB   0.135   90.097
   0.75   793530    PB   0.135    9.005
   1.25    79094    BB   0.134    0.898

TOTAL AREA =     881200
MUL FACTOR =   1.0000E+00
```

Typical HP 5890 Test Chromatogram (using an HP 3390A Reporting Integrator)

To access this function, the following key sequence is entered:

SIG 1 (or SIG 2) 9 ENTER

Test plot mode is confirmed by the display SIGNAL 1 (or 2) TEST PLOT. Pressing SIG 1 (or SIG 2) a second time displays the current signal level value (which is 0.0 initially). This permits monitoring the output signal.

The chromatogram is initiated by pressing START. The chromatogram continues to cycle until STOP is pressed. Each cycle is about 1•1/2 minutes in length.

Note:

- Setpoint values assigned to RANGE 2↑() and ATTN 2↑() affect level of the test signal output, as do equivalent functions on the receiving device.
- An oven temperature program (e.g., a setpoint value for RATE **other** than 0) must be set up at the HP 5890 for the test plot to function.

The test chromatogram is useful as a troubleshooting aid in deciding whether a lost or noisy signal observed at a connected integrating or chart recording device is due to a chromatographic problem (lost sample due to leaks, noise due to a dirty detector, etc.), versus problems either with the integrating/recording device itself, or in its connecting cables.

If the test chromatogram does not exhibit any problems at the integrating/recording device, a chromatographic problem is likely to exist; if the test chromatogram exhibits noise, or does not appear at all, the problem is **not** likely to be chromatographic.

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.

INET permits:

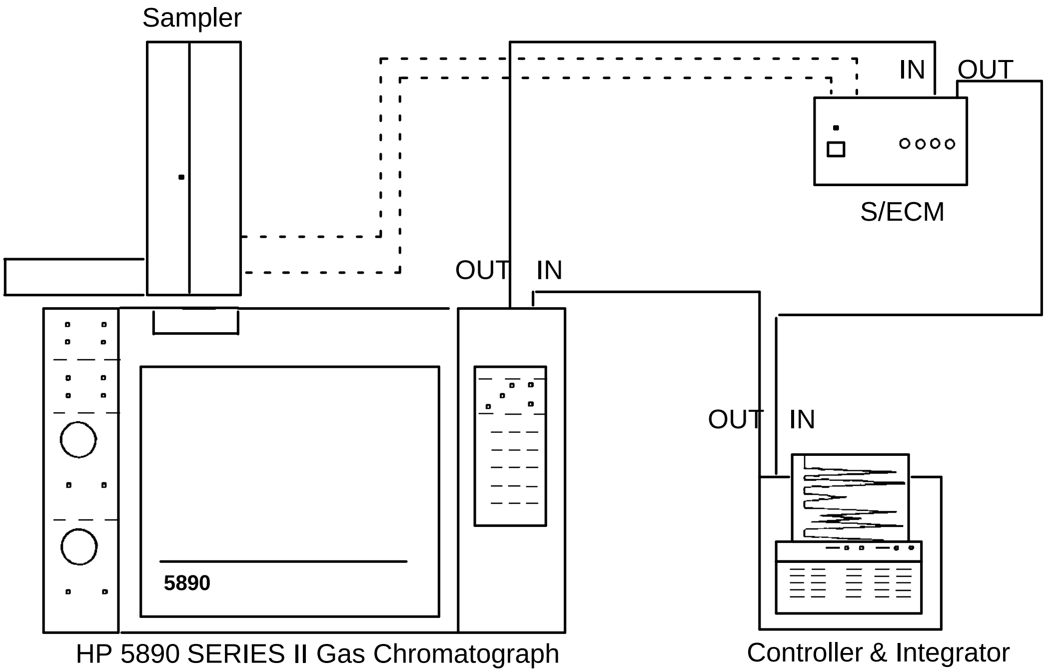
- Management of active workspace (described below) among instruments, a controller, and storage and print media.
- Sharing of run and readiness conditions among INET instruments.
- Sharing of setpoint and parameter information among INET instruments.
- Automation of data collection, sample tracking, and report generation.

Note: 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.

The controller

INET is a series•connected **loop** of devices, with the output of each device connected to the input of the next. A message sent from a particular device travels around the loop, returning to the same instrument as confirmation of transmission. Thus, the loop must be continuous, and all devices must be **on** for the loop to function.

Figure 5-6



Typical INET Loop

Each INET must have one (and only one) device defined as the **controller**. The **controller** is responsible for network configuration when the network is first connected and powered **on**.

The controller then retains this status for subsequent loop operations, maintaining its responsibility as network **traffic manager**. Note that the device defined as controller usually has other capabilities.

The controller assigns addresses to each device on the loop, and configures them to operate in orderly fashion. In addition, the controller provides user access to the network configuration so desired changes can be made.

For a typical analytical system, the default, automatic configuration should be acceptable in most cases. For specific information regarding configuration, consult appropriate manual(s) for the **controller** device (the HP 5890 is never a **controller**).

An instrument

Addresses

An **instrument** is a device, housing together a collection of functions, and having a single model number. It has a single pair (IN and OUT) of INET cable connections.

The INET controller assigns each instrument a unique address, in order, around the loop. Thus, addresses correspond to the physical order of connections around the loop.

Note that the **controller** usually is also an **instrument** in that it has capability of performing various analytical tasks (for example, integration).

Data or a command message destined for a particular instrument is labelled with the corresponding address for the instrument.

Setpoints

The controller passes setpoints among INET instruments (including itself), and to and from local storage. Setpoints are grouped according to the instrument to which they **belong**, and labelled with the corresponding INET address. Setpoint traffic is automatic, once appropriate commands are entered at the controller.

If an instrument receives setpoints intended for a different instrument because of an address error, it rejects them, causing the controller to print an error message.

Generally, each instrument provides a means for entering its own setpoints (i.e., a keyboard or control panel). The HP 19405A S/ECM is an exception: its setpoints are defined through the controller.

Except for the controller, each instrument handles setpoints for instrument(s) other than itself only as blocks of data to be transmitted, but not altered.

Active workspace

Each device in an INET loop provides storage area for its own setpoints and parameters. These individual storage areas (each containing setpoints and parameters for the specific instrument) are also available to any other device in the loop. For example, the controller may access them for listing and/or storage.

HP 5890 INET states

At the HP 5890, when a part of INET, the RUN LED provides indication of INET status:

- If the RUN LED is off, the INET system is in its **idle** state, waiting for initiation of some action (e.g., starting a run, listing information, etc). In this state, the HP 5890 NOT READY LED is turned on if the HP 5890 is not ready, and/or if the INET system itself is not ready (e.g., if a device on the loop is busy). A message SYSTEM NOT READY will appear on the HP 5890 if some other device on the INET loop reports **not ready**, **and** the HP 5890 is itself **ready**.

  CLEAR may be pressed to display things that are not ready.

- If the RUN LED is blinking, the INET system is in pre•run or post•run operation (e.g., a sampler cycling, a report in progress, etc). In addition, either SYSTEM IN PRE-RUN or SYSTEM IN POST-RUN is displayed if CLEAR is pressed. The NOT READY LED is on **only** if the HP 5890 itself is not ready.

- If the RUN LED is on, a run is in progress. The NOT READY LED is turned on **only** if the HP 5890 itself should become not ready.

INET operation

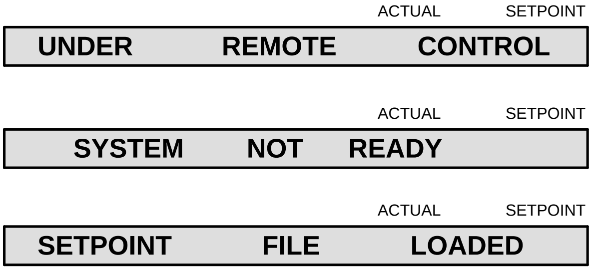
In using the INET function, chromatographic parameters are entered normally through the HP 5890 keyboard. Integration parameters are entered at the controller. Parameters for other devices on the INET loop may be entered at the controller, or at their own keyboards. Collectively, the separate sets of parameters constitute a single set of parameters for an **analysis**.

The intent here is to discuss INET operation **only** from the point of view of the HP 5890. For specific information regarding INET loop control (configuration, workfile storage and recall, etc), consult the appropriate operation manuals for the **INET controller** device. Also consult appropriate manual(s) for other devices (sampler/event control module, etc) configured in the loop.

Typical displays occurring when the HP 5890 is under **INET** control are shown in Figure 5•7.

Figure 5-7

INET Displays



Typical INET Loop Displays

- Keyboard setpoint entry is not allowed at the HP 5890 while a workfile or method is stored or listed at the controller: UNDER REMOTE CONTROL is displayed if an entry is attempted.

If a setpoint entry at the HP 5890 keyboard is in progress when a workfile or method is stored or listed at the controller, the entry is aborted. After the operation finishes, the HP 5890 returns to the same setpoint display.

- When a stored workfile or method is recalled to active workspace at the controller, its setpoints are automatically downloaded into devices on the INET loop, including the HP 5890. Successful download at the HP 5890 is confirmed by the display, SETPOINT FILE LOADED. Any instrument function key (e.g., OVEN TEMP), or CLEAR , erases the message.

  If a setpoint entry at the HP 5890 keyboard is in progress when a workfile or method is recalled, the entry is aborted.

- If problems occur in transferring chromatographic setpoints to the HP 5890, the HP 5890 retains its present setpoints, so there is no indication at the HP 5890 that transfer was attempted. Appropriate error messages are printed at the controller device.
- SYSTEM NOT READY, if displayed, indicates one or more devices on the INET loop report **not ready**.
- UNDER REMOTE CONTROL is displayed if, from a host computer system, a command has been entered to lock the HP 5890 keyboard to prevent alteration of setpoints, and entry of a setpoint value is attempted at the HP 5890.
- In case of INET system failure, the HP 5890 may be operated by itself (INET cables should be disconnected at the HP 5890). If, as a result of the failure, the HP 5890 keyboard does not respond normally to key presses, switch off the instrument at its main power switch. Upon restoring power, the keyboard should be active.

Automatic INET reconfiguration

In the following circumstances, INET automatically reconfigures under direction of the controller:

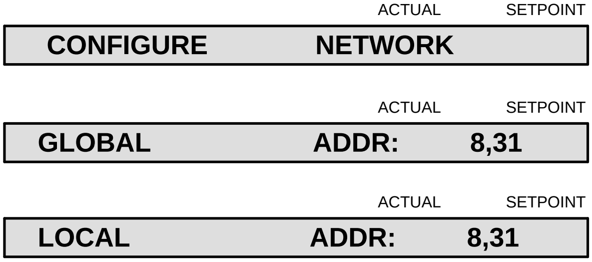
- Recovery from a power failure.
- Recovery from any particular device on the loop being switched off, then on again.
- Recovery from a disconnected (then reconnected) loop cable.

Consult appropriate manuals for the controller in the event of problems arising from any of these circumstances.

INET configuration

The CONFIGURE NETWORK function provides four features: verifying the INET address for the HP 5890 (as determined through automatic loop configuration), setting the default HP•IL address to be used when the HP 5890 is connected to some device where addresses must be set manually (i.e., no automatic loop configuration), switching the INET function at the HP 5890 between **global** or **local**, and verifying INET signal definitions. Each feature is discussed separately.

Figure 5-8



```
                        ACTUAL      SETPOINT
 CONFIGURE           NETWORK

                        ACTUAL      SETPOINT
 GLOBAL              ADDR:     8,31

                        ACTUAL      SETPOINT
 LOCAL               ADDR:     8,31
```

CONFIGURE NETWORK Displays

Figure 5•8shows displays resulting from the key sequence:

CLEAR ▪ 3 ENTER

Switching between Global and Local

With regard to the INET function at the HP 5890, there are two operating modes: **global** or **local**. In **global** mode (default mode), HP 5890 START and STOP keys, when pressed, affect other devices on the INET loop. In **local** mode, however, pressing START or STOP at the HP 5890 affects only the HP 5890. A run may be started or stopped at the HP 5890 without affecting other devices on the INET loop.

In **local** mode, note that the HP 5890 remains part of the INET system; it reports its **readiness** to the system and pressing START and STOP keys on other devices on the INET loop (e.g., the controller) will affect HP 5890 operation.

Once in CONFIGURE NETWORK, pressing ON or OFF switches, respectively, between **global** or **local** mode shows resulting displays as in Figure 5•9.

Figure 5-9

```
                          ACTUAL      SETPOINT
 GLOBAL              ADDR:        8,31

                          ACTUAL      SETPOINT
 LOCAL               ADDR:        8,31
```

INET GLOBAL/LOCAL Displays

An example of where having the HP 5890 in **local** mode might be useful is in the case of conditioning a column: the HP 5890 may be started or stopped as desired without affecting other devices on the INET loop.

Note that **global** mode has two states: if GLOBAL flashes (default mode) when displayed, the HP 5890 is in **global** mode, but **not** configured into the INET system. When the HP 5890 is properly configured into the INET system, GLOBAL is displayed continuously. This feature provides a convenient diagnostic to determine if system configuration has occurred (at least as far as the HP 5890 is concerned).

INET/HP-IL addresses

Figure 5•10 shows displays occurring either in verifying an INET address set through automatic loop configuration or in entering a specific HP•IL default address used when the HP 5890 is included in an HP•IL loop without automatic configuration. The address is maintained in battery•protected memory along with other instrument setpoints.

Figure 5-10

```
                                  ACTUAL      SETPOINT
 GLOBAL                ADDR:        8,31

                                  ACTUAL      SETPOINT
 DEFAULT      ADDRESS        *       25

                                  ACTUAL      SETPOINT
 GLOBAL                ADDR:        8,25

                                  ACTUAL      SETPOINT
 ADDRESS      LIMIT        =      31
```

INET/HP-IL ADDR: Displays

Verifying the HP 5890 INET address

In Figure 5•10, note the two numeric values following ADDR:. The first of these numbers is the INET address for the HP 5890, determined via automatic loop configuration.

The **specific** number shown depends upon how INET cables are connected among devices included in the loop. The value shown in the example (8) implies the HP 5890 is the first instrument on the loop, starting from the OUT receptacle on the controller device (the controller is always defined as 0). A 9 indicates the HP 5890 is the second device on the loop, etc, to a maximum value of 31.

If cabling is altered, or if one or more devices are powered off and then on again, automatic loop configuration, initiated by the controller, updates the displayed value accordingly.

Setting the default HP-IL address

Remaining displays in Figure 5•10 show the process of defining a specific HP•IL address for the HP 5890. Entry of any value from 8 through 31 is permitted. An attempt to enter an invalid value results in the ADDRESS LIMIT message shown.

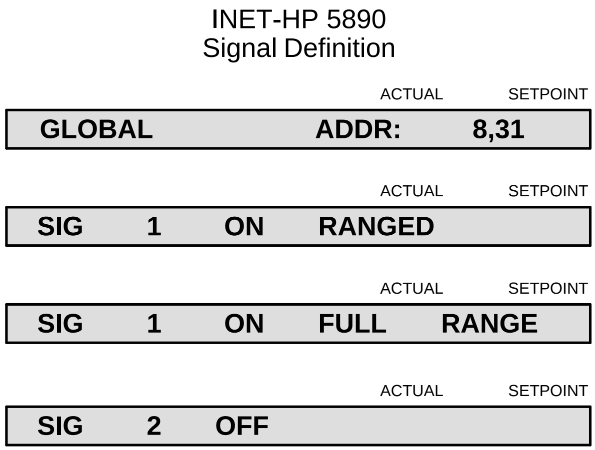
INET-HP 5890 signal definition

INET signal definition (defined **at the controller**) may be verified at the HP 5890. To display the definitions, enter:

CLEAR • 3 ENTER
(SIG 1 or SIG 2)

Figure 5•11 shows resulting displays.

Figure 5-11



INET-HP 5890
Signal Definition

```
                                   ACTUAL      SETPOINT
GLOBAL                 ADDR:       8,31

                                   ACTUAL      SETPOINT
SIG      1      ON     RANGED

                                   ACTUAL      SETPOINT
SIG      1      ON     FULL       RANGE

                                   ACTUAL      SETPOINT
SIG      2      OFF
```

INET Signal Definition Displays

From the displays, the following may be noted:

- HP 5890 signal channels are designated SIG 1 or SIG 2.
- ON indicates the given signal channel is considered active by the controller; data from this signal channel is transmitted to other devices on the INET loop.

  Similarly, OFF indicates the channel is considered inactive; no data from this signal channel is transmitted to other devices on the loop.

  It is important to note that ON or OFF in this context are strictly INET definitions, defined at the system controller. They do not, for example, bear any relationship to whether or not a given detector assigned to the signal channel is turned on or off.

- RANGED versus FULL RANGE indicates the dynamic range for the data to be transmitted to other devices on the loop; dynamic range for RANGED data is set at the HP 5890 according to the setpoint for RANGE 2↑(). Dynamic range for FULL RANGE data is limited only by the detector itself. The choice of the type of data to be transmitted is set at the controller.

HP-IL loopback test

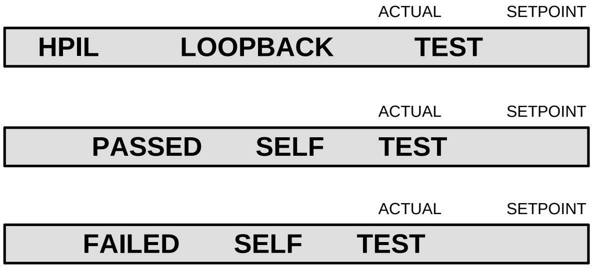
The HPIL LOOPBACK TEST may be performed any time to verify that HP 5890 INET communication is performing satisfactorily. Testing involves setting up the HP 5890 to send an INET message directly to itself by connecting its INET output to its INET input. The following procedure is used:

1. Disconnect INET cables at their respective INSTRUMENT NETWORK IN and OUT receptacles on the HP 5890 (located beneath the top right cover panel).
2. Choose either one of the cables and disconnect it at the next device on the INET loop.
3. Connect this free cable to both IN and OUT INSTRUMENT NETWORK receptacles on the HP 5890.
4. Enter the following key sequence:

CLEAR • 7 ENTER

Upon pressing ENTER, the test is performed: the HP 5890 both sends and verifies a diagnostic message to itself through the connected cable. Each press of ENTER repeats the test. Each test requires about one second. Figure 5•12shows displays to be expected.

Figure 5-12



HPIL LOOPBACK TEST

PASSED SELF TEST

FAILED SELF TEST

HPIL LOOPBACK TEST Displays

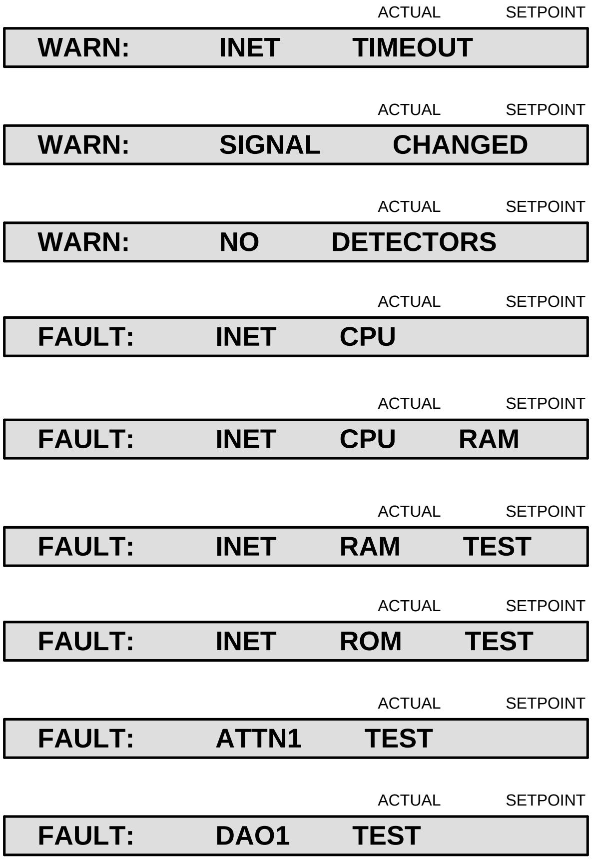
The message PASSED SELF TEST indicates INET, at least with respect to the HP 5890, is performing satisfactorily. If FAILED SELF-TEST is displayed, a bad cable may be indicated; install a different INET cable and repeat the test. If FAILED SELF-TEST is displayed again for a second cable, electronic problems within the HP 5890 are indicated.

Note: The **loopback test** may be used to check for continuity in an INET cable; an open cable causes test failure. Verify that the cable is at fault (rather than the HP 5890) by testing a second cable.

If an intermittent cable problem is suspected, the test may be repeated as necessary while flexing the cable (particularly at its plugs). An ohmmeter should also be used to test for problems; it is a reliable method for testing continuity.

Warn: and fault: messages

Figure 5-13



WARN: INET TIMEOUT

WARN: SIGNAL CHANGED

WARN: NO DETECTORS

FAULT: INET CPU

FAULT: INET CPU RAM

FAULT: INET RAM TEST

FAULT: INET ROM TEST

FAULT: ATTN1 TEST

FAULT: DAO1 TEST

Signal Control WARN: and FAULT: Messages

Figure 5•13shows possible WARN: and FAULT: messages associated with signal functions. In general, the following problems are indicated if the following messages appear:

- WARN: INET TIMEOUT is displayed if information transmission on the INET loop is interrupted; for example, if an INET loop cable is accidentally disconnected.

- WARN: SIGNAL CHANGED and/or WARN: NO DETECTORS is displayed if a detector previously assigned to a particular signal channel is found to be absent; for example, if the signal board for a given detector should fail or be removed for service.
- FAULT: INET CPU is displayed if the processor (and/or its associated circuitry) for HP 5890 INET operations should fail.

HP 5890 diagnostics generating the above message displays are active at all times in normal operation. The next group appears only if a problem is found to exist at HP 5890 power•on:

- Remaining FAULT: INET messages are displayed if problems are diagnosed in INET circuitry.
- FAULT: ATTN1 TEST and/or FAULT: DAC1 TEST occur if problems are diagnosed in signal path circuitry for a particular signal channel (channel 1, in the example displays).

In general, if signal problems are suspected, power to the HP 5890 may be turned off, and then on again to perform internal self•testing. Appropriate message displays occur if problems are found to exist.

Note: In case multiple problems exist simultaneously, press CLEAR to **roll** through NOT READY and/or FAULT: displays.

Note that, with exception of WARN: OVEN SHUT OFF, CLEAR erases any displayed WARN: message.

For WARN: messages, the instrument remains operational, except for the function indicated by the message: the message is erased upon pressing any instrument function key (e.g., OVEN TEMP). For FAULT: messages, in addition to the message, the red NOT READY LED blinks: the instrument CANNOT be operated until the problem is corrected.

If a WARN: or FAULT: message occurs in normal operation, try switching power to the instrument off, and then on again. If the problem recurs when power is restored, service procedures are indicated.

File compatibility with data handling devices

You must have the HP 5890 SERIES II in the proper mode for file compatibility with your data handling device.

What are the modes?

There are 2 file transfer modes: HP 5890A and HP 5890 SERIES II.

HP 5890A File Transfer Mode	HP 5890 SERIES II File Transfer Mode
Transmits HP 5890 setpoints:	Transmits HP 5890 setpoints plus: Timetable events Inlet pressure and temperature programs Up to 450° oven and FID temperature Cryo Blast and Ambient setpoints TCD sensitivity Aux temperature setpoint

What is the proper mode for my data handling device?

Selection of the proper mode depends on the product and version of your software.

How do I know in which mode my GC is configured now?

1. Turn power **off**, then **on**.
2. Check the GC display.

Figure 5-14

ACTUAL SETPOINT

HP 5890A mode **EMULATION MODE OK**

ACTUAL SETPOINT

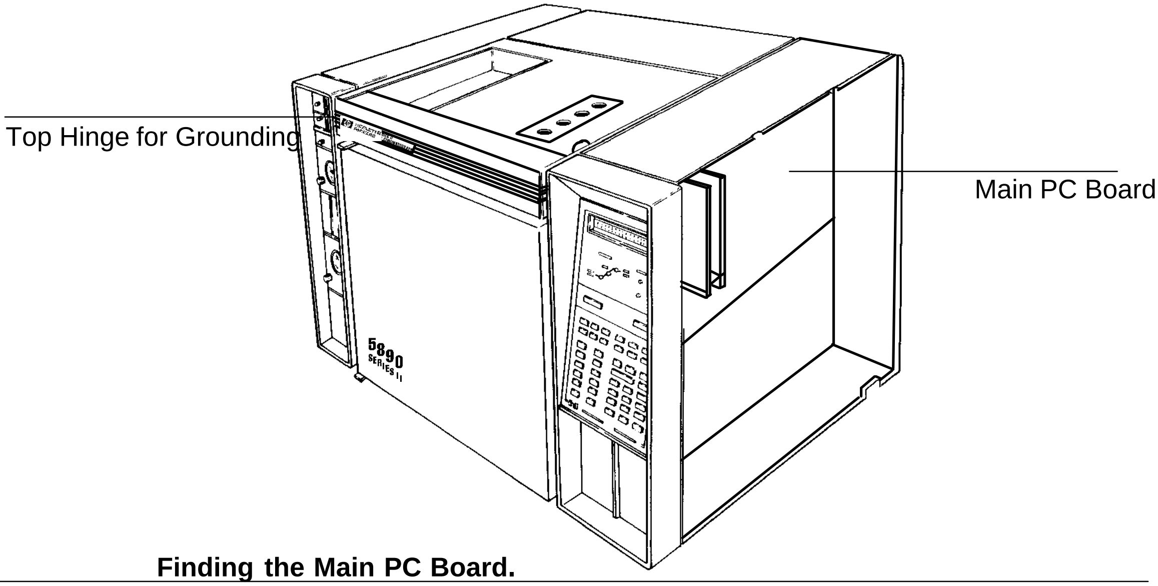
HP 5890 SERIES II mode **PASSED SELF TEST**

GC Displays for File Transfer Modes

How do I change modes?

1. Turn power **off**.
2. Remove the GC side panel, and locate the main PC board.

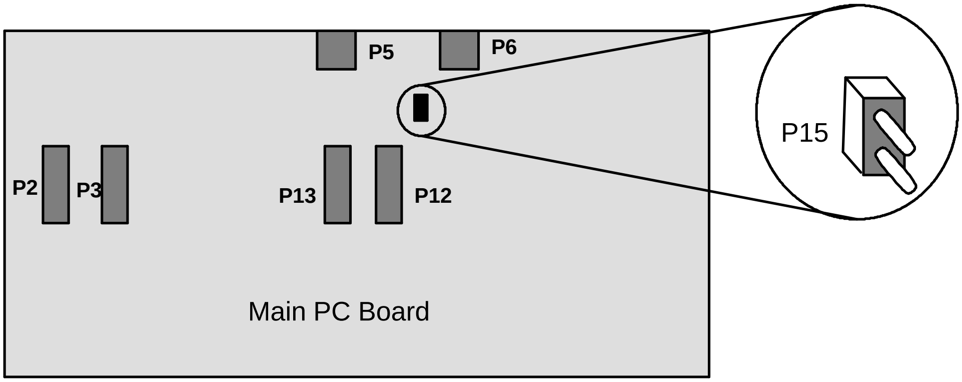
Figure 5-15



Finding the Main PC Board.

3. Find component P15 on the main PC board.

Figure 5-16



Finding component P15 on the Main PC Board.

4. Set the jumper (Part No. 1258•0141)for the proper mode. To avoid electrostatic damage to the main board, ground yourself to the GC chassis with an ESD strap, or touch an unpainted area of the oven such as the door hinge.

Figure 5-17

Setting the jumper.

How to convert HP 339X Integrator workfiles from 5890A to SERIES II mode:

1. Turn the GC **off**.
2. Follow the previous instructions to set the GC for 5890A mode (use proper grounding).
3. Download the workfile from the integrator.
4. Turn GC power **off**.
5. Remove the P15 jumper. (Now the GC is in SERIES II mode.)
6. Turn GC power **on**.
7. Add SERIES II setpoints (time table, etc).
8. Store the workfile at the integrator.

Note: Using STORE on the HP 5890 SERIES II, up to 4 integrator workfiles can be converted with each jumper change sequence.

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6

Inlet Systems

Inlet Systems

This chapter provides information for the following HP 5890 SERIES II (hereafter referred to as HP 5890) inlet systems:

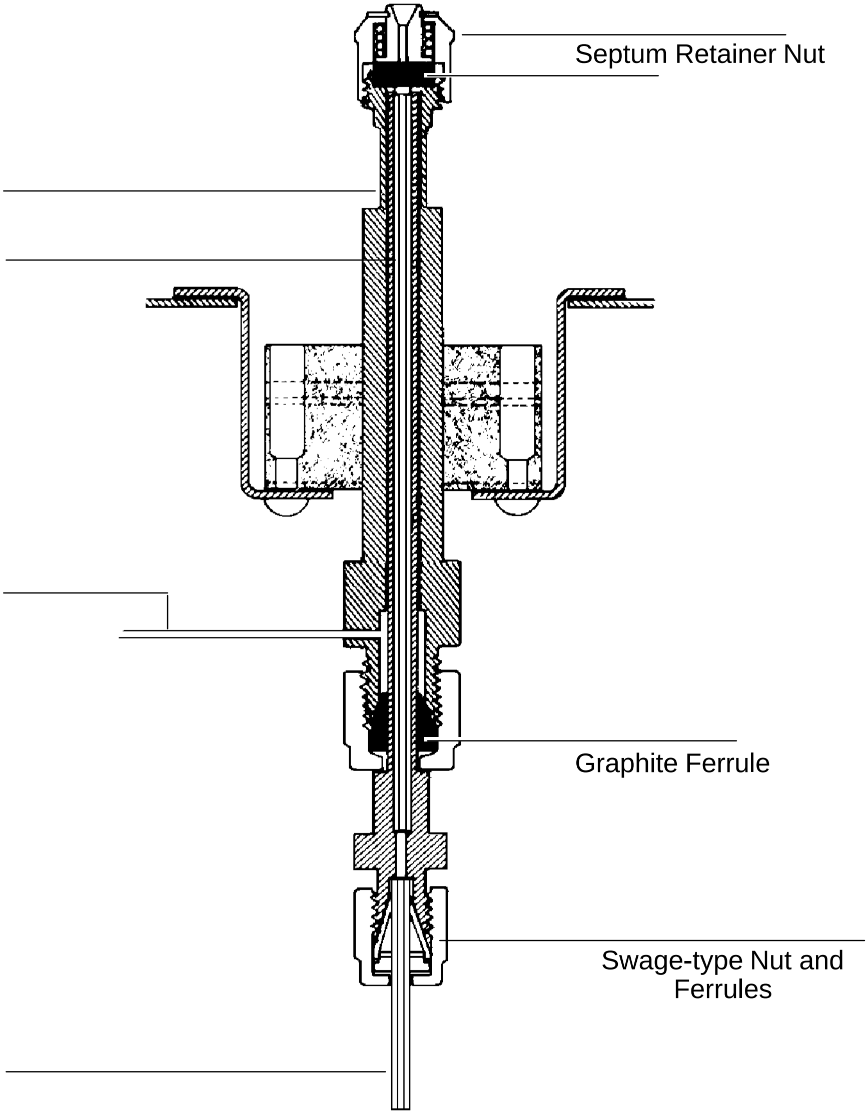
- Packed column inlet
- Septum•purged packed column inlet
- Split/splitless capillary inlet

For cool on•column information, see the manual *Programmable Cool On•Column Inlet.* Maintenance information is provided in Chapter 8**,** *Preventive Maintenance.*

Packed column inlet

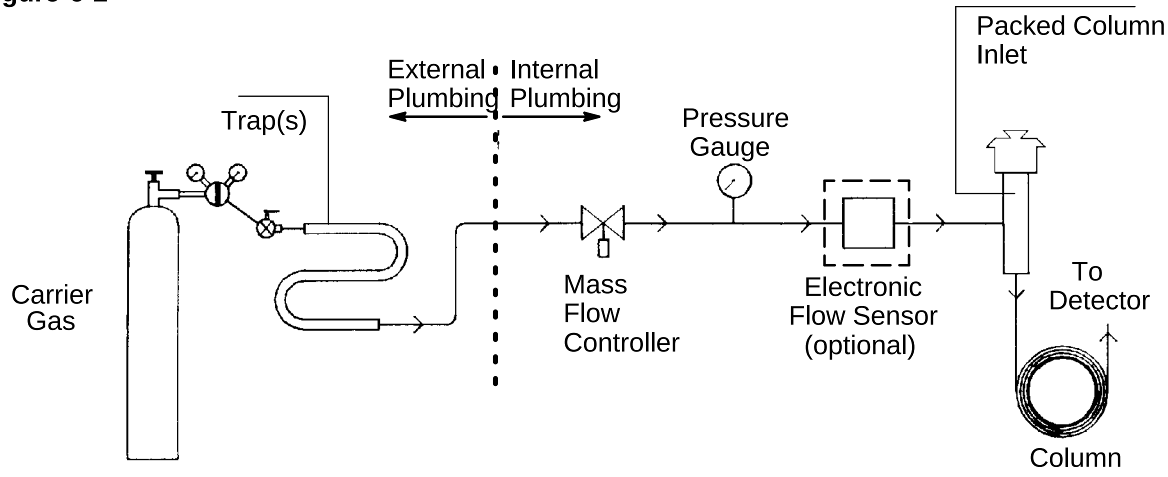
The packed column inlet may be used with HP Series 530 µ capillary columns, metal packed or glass packed columns. Additionally, on•column injection is possible with 1/4•inch glass packed columns. The packed column inlet is optimized for low to intermediate temperatures (200-300 °C).

Figure 6-1



Packed Column Inlet

Figure 6-2



Flow Diagram, Packed Column Inlet (with electronic flow sensor)

Liquid sample is rapidly volatilized inside the inlet. To ensure complete volatilization, inlet temperature typically should be at least 20°C greater than the highest oven temperature to be used.

Assuming column and liner (plus insert, if used) are properly installed and the system is leak•free,the following is information necessary for proper operation.

Electronic flow sensor

If Electronic Flow Sensing (EFS) is installed in the carrier gas system to the inlet, total **supply** flow rate through the system may be displayed (see Chapter 4**,** *Electronic Flow Sensing*, for details). This is particularly convenient in situations when carrier flow must be turned off (as when changing gas supplies, or if supplies must be turned off nightly, weekends, etc.).

Note: The EFS requires at least one hour for thermal equilibration for reliable readings.

Assuming the system to be leak•free(and if **total** flow is < 200 ml/min), after setting the desired column flow rate, total flow through the system should be noted via the EFS.

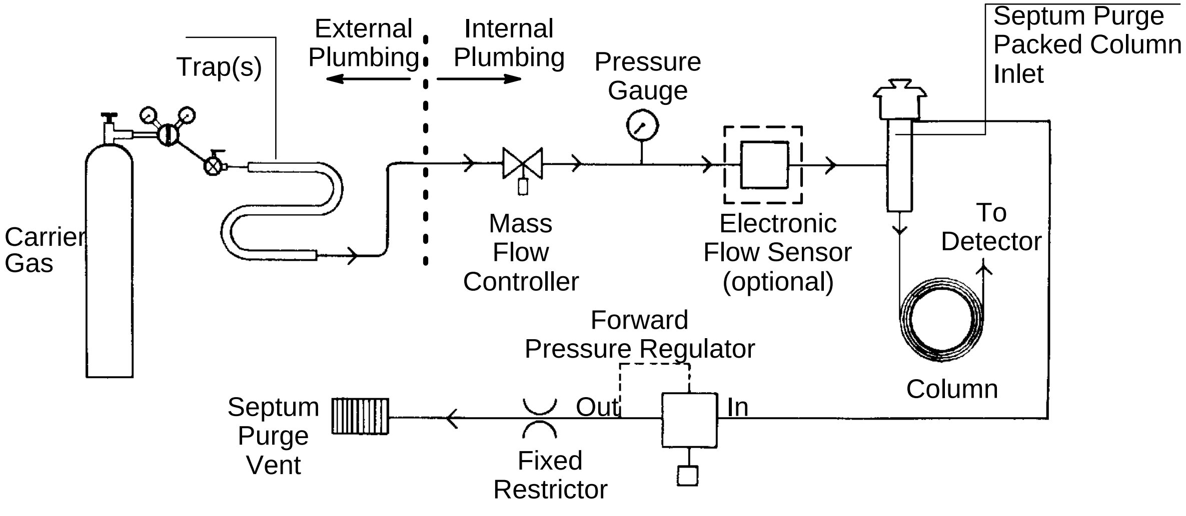
The original column flow rate is reestablished simply by adjusting the mass flow controller so the original total system flow rate value is displayed again.

Septum-purged packed column inlet

The septum•purgedpacked column inlet may be used with HP Series 530 μ capillary columns, metal packed or glass packed columns. Additionally, on•columninjection is possible with 1/4•inchglass packed columns.

The septum•purgedinlet provides superior reproducibility and quantitative accuracy at high inlet temperatures (250 to 400°C).

Figure 6-3



Flow Diagram, Septum-Purged Packed Column Inlet

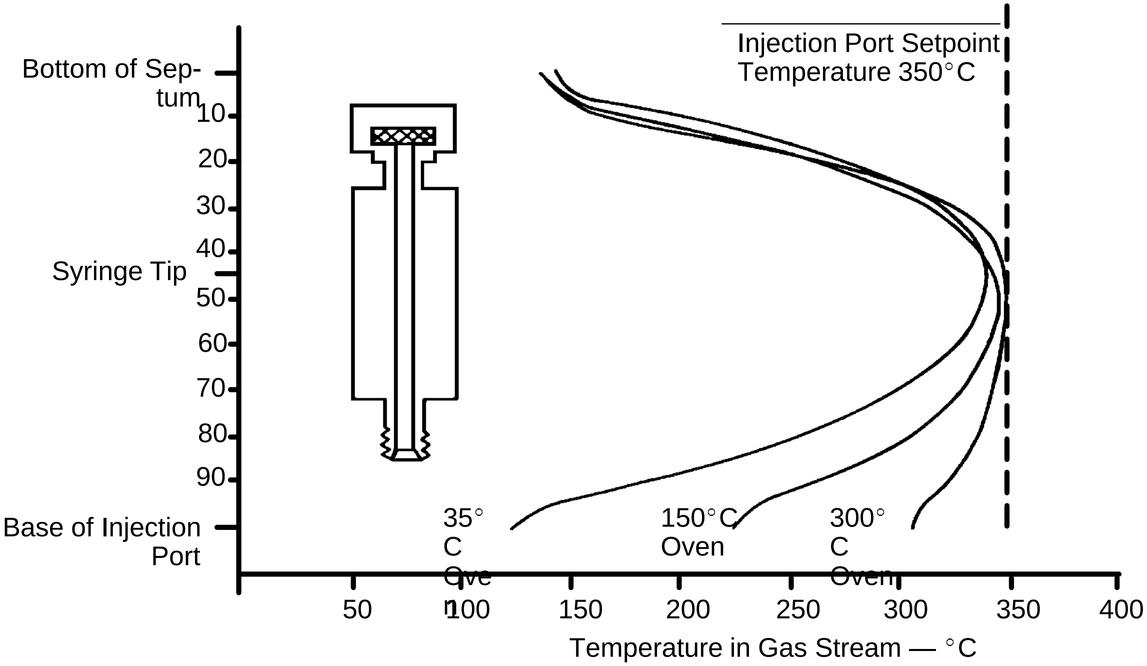
Problems at high inlet temperatures

A common problem with conventional packed column inlets operated at high temperatures is septum bleed and the associated ghost peaks. To minimize this effect, some inlet systems are designed with steep temperature gradients throughout the entire upper length of the inlet to provide a **cool** septum and minimal ghost peaks. However, this temperature gradient allows condensation of high boiling point compounds resulting in nonreproducibility and discrimination against these compounds. (This effect has sometimes been mistakenly called needle fractionation.)

A thermally optimized high-temperature inlet

The septum•purgedpacked column inlet has been thermally optimized to provide an extremely uniform thermal profile throughout the flash vaporization region. This thermal profile remains very close to the inlet setpoint temperature throughout most of the gas stream. Then, up in the purge region of the inlet, a very steep temperature gradient is forced with a large cooling fin. This results in longer septum life, and reduced ghost peaking when run in a nonpurging mode.

Figure 6-4



Thermal Profiles

This optimized thermal profile allows very reproducible results and virtually eliminates injection port discrimination against high boiling point components. When combined with fast automated injection, excellent quantitative accuracy is possible.

Septum purge

Best results are obtained by running a very low septum purge flow (1 to 2 ml/min. of purge flow is recommended• never more than 10% of column flow rate). Once the purge flow has been set, it will remain constant, regardless of changes in column head pressure or column flow rate. An alternate septum nut is provided which allows operation without purge flow if desired.

When operating the inlet with septum purge, low bleed septa are unnecessary and the selection of septa should be made primarily for good sealing and extended septa life reasons.

On a periodic basis (every 1 to 2 months), the Teflon•coatedO•ring sealing the purge cavity should be replaced.

Electronic flow sensor

If Electronic Flow Sensing (EFS) is installed in the carrier gas system to the inlet, total **supply** flow rate through the system may be displayed (see Chapter 7**,** *Electronic Flow Sensing*, for details). This is particularly convenient in situations when carrier flow must be turned off (as when changing gas supplies, or if supplies must be turned off nightly, weekends, etc.).

Note: The EFS requires at least one hour for thermal equilibration for reliable readings.

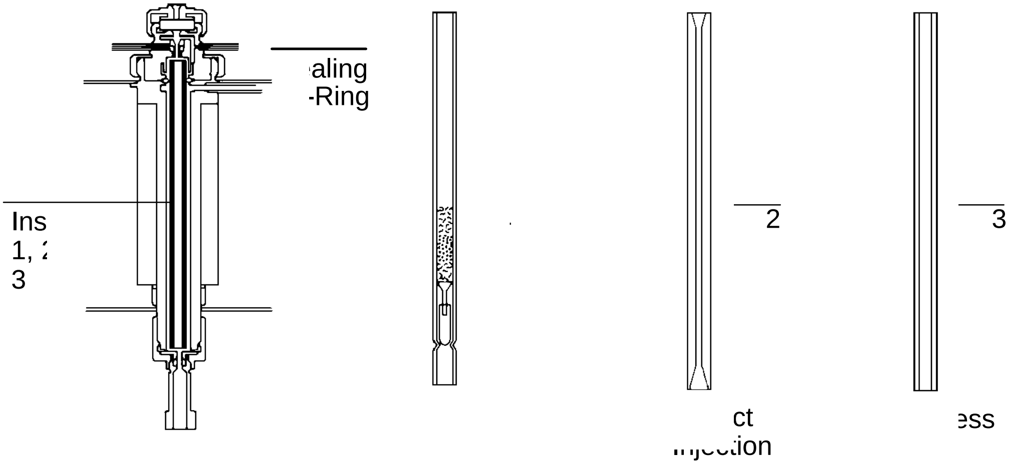
Assuming the system to be leak•free(and if **total** flow is < 200 ml/min), after setting the desired column flow rate, total flow through the system should be noted via the EFS.

The original column flow rate is reestablished simply by adjusting the mass flow controller so the original total system flow rate value is displayed again.

Split/splitless capillary inlet

Figure 6-5

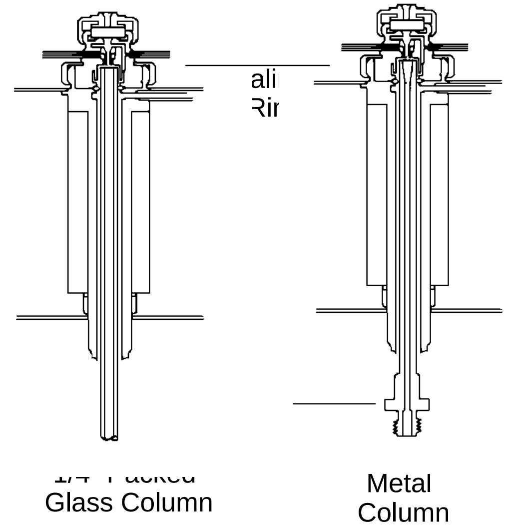
A. CAPILLARY COLUMN



B. PACKED COLUMN

Item No.	Description	Part Number	
1	Split Insert (packed)	18740-60840	
1	Split Insert (unpacked)	18740-80190	
2	Direct Injection Insert	18740-80200	
3	Splitless Insert	18740-80220	Available for Purcha
4	1/8-in. Column Insert	18709-80030	
5*	1/4-in. Column Insert	18745-80010	

*NOTE: Not shown



Split/Splitless Capillary Inlet

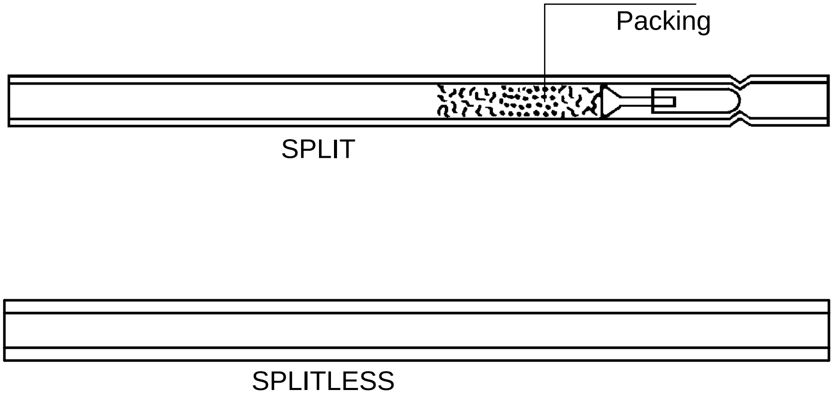
The multiple•modesplit/splitless capillary inlet system may be used with any of the common types of capillary columns (fused silica, quartz, glass, metal).

Specific sampling modes include:

- Split, for major•componentanalyses.
- Purged splitless, for trace•componentanalyses.

Each mode requires installation of a specific inlet insert.

Figure 6-6



Available Inlet Inserts

Note that performance in capillary analyses is closely related to the insert used (and sampling mode). Inserts supplied are **average**, suitable for good performance over a variety of general applications. For specialized applications, however, to optimize inlet performance, custom•madeinserts may be used: any custom•madeinsert must not exceed 6.52 mm od, and must be 78.5 ± 0.1 mm in length to operate properly.

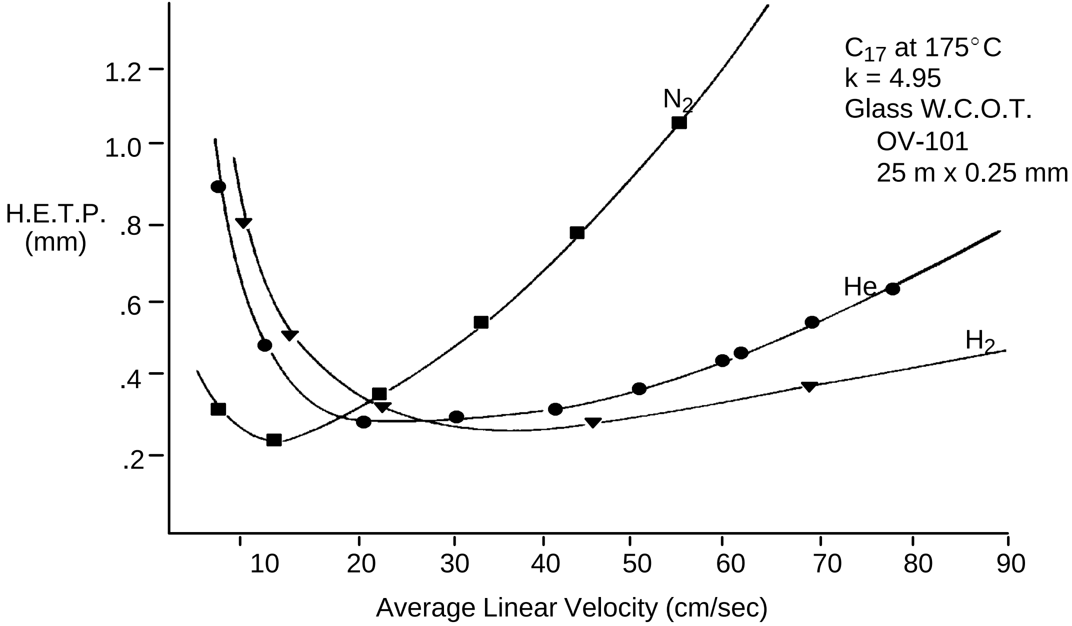
Carrier gas considerations

Carrier gas choice, and its average linear velocity through the column, strongly influences column efficiency, analysis time, and system stability.

In general, the carrier gas is chosen to maximize component resolution and detector performance while minimizing overall analysis time.

Figure 6•7,a family of van Deemter curves for common carrier gases, illustrates the effect of gas choice and linear velocity (µ) on column efficiency (HETP, Height Equivalent to a Theoretical Plate) for a particular column and analysis.

Figure 6-7



H.E.T.P. versus Linear Velocity (µ)

Where temperature programming is to be performed, a linear velocity value should be chosen in the region 1•1/2to 2•1/2times the linear velocity at the minimum point on the HETP curve for the particular carrier gas. This minimizes change in column efficiency over the temperature range used.

Where column efficiency is not an important consideration, or in isothermal operation, any reasonable linear velocity value may be chosen.

Van Deemter curves demonstrate advantages of using either He or H_2 as carrier gas. From the curves, several observations may be made:

- Minima for He and H_2 occur at much higher average linear velocities than N_2. Thus, He, or even better, H_2, can be used at far higher velocities than N_2 with only small loss in efficiency. Use of H_2 or He allows shorter overall analysis times.
- An additional benefit in using H_2 is that high velocities can be achieved at relatively low column head pressures.
- For any given compound, the minimum in the N_2 curve is quite pronounced; consequently, there is only a small linear velocity range over which column efficiency is maximized.
- H_2, and He to a lesser extent, exhibit relatively flat minima. Both are better suited as carrier gases when temperature•programmed analyses are performed.

An unrelated problem with N_2 is that it is available in many grades and is often contaminated with water and/or light hydrocarbons. He and H_2 are typically available in higher purities.

Overall, H_2 is the best carrier gas choice, with He nearly as good.

Initial column head pressure

In using capillary columns, linear velocity through the column must be set. This, in turn, is controlled by pressure at the head of the column. Pressure required to obtain a particular velocity depends primarily upon bore (ID) and length of the particular column and oven temperature.

Hewlett•Packardfused•silicacapillary columns may be conveniently broken into several categories based upon their bores. Table 6•1lists suggested **initial** pressures for some capillary column bores and lengths.

Table 6-1. Suggested Initial Column Pressures (kPa) for Various Capillary Column Bores and Lengths

Nominal ID (mm)	Nominal Length (m)		
	12	**25**	**50**
0.20	135	223	347
0.32	45	82	137
0.53	11	23	42

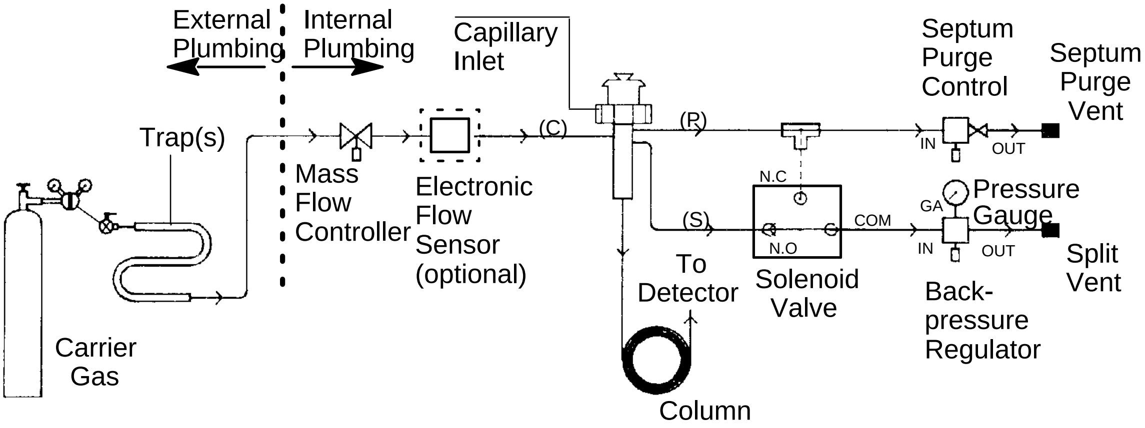
It must be emphasized that values in this table are recommended as starting points **only**!
Values listed are independent of carrier gas used.

It is important to note that flow settings made for one particular column are not necessarily correct for any other column, or for every application. Column flow rate should be verified, and adjustments made as necessary, whenever the column or other parts of the flow system are changed.

Split sampling

WARNING In performing split sampling, when using hazardous chemicals, and/or H_2 carrier gas, effluent from the split vent should be vented to a fume hood or appropriate chemical trap.

Figure 6-8



Flow Diagram, Split Operation

Due to short sample residence time inside the inlet, the technique requires rapid volatilization; thus, inlet temperature must be high enough to ensure this.

The backpressure regulator in the split vent path maintains constant pressure at the head of the column. Total inlet flow, controlled by a mass flow controller, divides between a septum purge path and flow down the inlet insert.

Flow through the insert is divided again, between flow into the column and flow around the bottom of the insert, up between the outside of the insert and inlet body, and on to the split vent.

The **split ratio** is defined as:

$$\text{Split Ratio} = \frac{\text{Split Vent Flow Rate + Column Flow Rate}}{\text{Column Flow Rate}}$$

where flow rates are volumetric, measured in ml/min.

The split ratio is an indicator of the fraction of total sample entering the column: the higher the value, the less sample enters the column.

For setting flow for split sampling, see Chapter 4 of the *HP 5890 Operating Manual.*

Verifying inlet purge status

Verify that inlet purge flow is currently on, and will remain on throughout runs to be made in split sampling mode.

- To display current inlet purge status:

  PURGE/VALVE A (or B)

  If OFF is displayed, press ON to restore inlet purging.

- To display elapsed time during a run when inlet purging will be halted:

  PURGE/VALVE A (or B) TIME OFF

- To display elapsed time during a run when inlet purging will be restored:

  PURGE/VALVE A (or B) TIME ON

  (or ON alone if PURGE TIME is already displayed)

For split sampling, both times should be set to 0.00 and inlet purging must currently be ON. If necessary, this is done simply by pressing 0 ENTER when PURGE TIME OFF (or ON) is displayed.

Splitless sampling

For splitless operation, the dilute sample is vaporized inside the inlet insert. Most of the sample is then swept onto the column.

For full column efficiency, vaporized sample components must **reconcentrate** at the head of the column prior to separation; without reconcentration, peak widths of eluting components reflect inlet insert volume rather than column efficiency. Reconcentration occurs via two processes, the **solvent effect** or **cold trapping**; these are both discussed on the next page.

Since the technique requires flow through the inlet insert to enter the column during injection, solvent vapor throughout the inlet continues to enter the column even after sample component reconcentration. This can create a long, substantial solvent tail, obscuring peaks of interest. Thus, after a short time interval, the inlet insert is purged of solvent vapor.

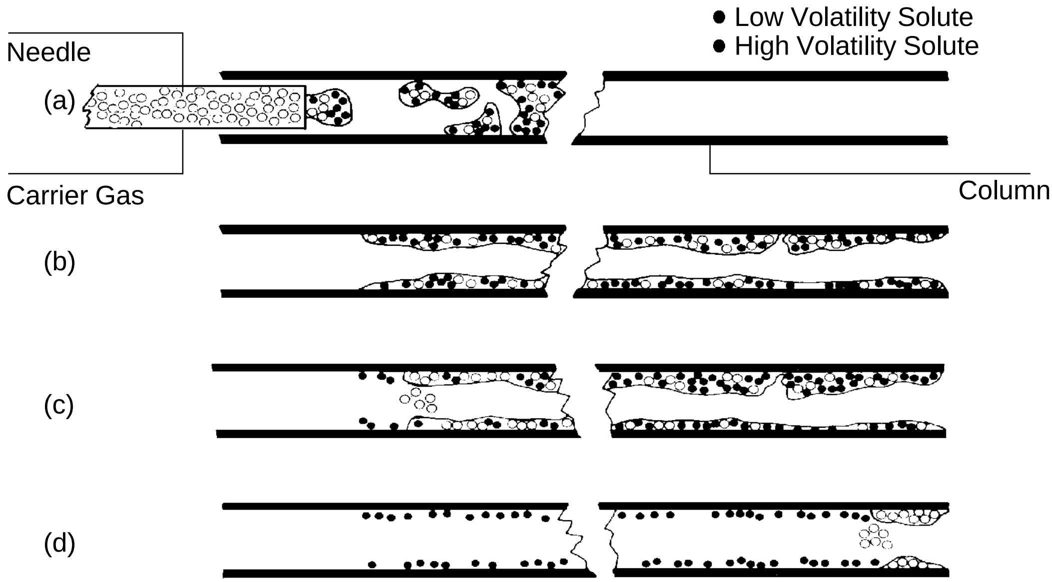
After the inlet insert is purged, oven temperature is increased, causing solvent trapped at the head of the column to volatilize and move through the column. Sample components are thus released to be separated.

Since sample residence time inside the inlet is longer than in split sampling, the inlet may be operated at a lower temperature: 150 to 200°C is adequate for most applications.

Solvent effect

One mode of sample reconcentration is the solvent effect which requires vaporized sample components to be retained by a region of stationary phase and absorbed solvent at the head of the column. In this region, the front of the sample **plug** undergoes stronger retention than does the rear: solvent•saturatedstationary phase acts as a barrier to sample components, thereby reducing their bandwidths.

Figure 6-9



The Solvent Effect

The solvent effect is described in great detail elsewhere: see Grob, K. and Grob, K., Jr., *Journal of Chromatography*, 94, page 53 (1974); Grob, K. and Grob, G., *Chromatographia*, 5, page 3 (1972).

To reconcentrate sample components via the solvent effect, oven temperature must be low enough so solvent remains at the head of the column for a sufficiently long time period. A good guideline is to have the column 10 to 30°C below the solvent boiling point. Table 6•2gives boiling points for some common solvents, along with suggested initial oven temperature ranges to use the solvent effect efficiently.

Table 6-2. Some Common Solvents: Suggested Initial Oven Temperatures to Use the Solvent Effect

Solvent	Boiling Point (°C)	Suggested Initial Oven Temperature Range (°C)
Diethyl Ether	36	10 to 25
n-Pentane	36	10 to 25
Methylene Chloride	40	10 to 30
Carbon Disulfide	46	10 to 35
Chloroform*	61	25 to 50
Methanol*	65	35 to 55
n-Hexane	69	40 to 60
Ethyl Acetate*	77	45 to 65
Acetonitrile	82	50 to 70
n-Heptane	98	70 to 90
i-Octane	99	70 to 90
Toluene	111	80 to 100

---

* Should be used **only** with cross-linked stationary phases.

The best solvent for a given application is found by trial and error, depending upon sample solubility and volatility, column polarity and type of stationary phase, and detector selectivity/sensitivity.

Note that, because of the solvent effect, retention times for the same components, but dissolved in different solvents, may be different: the solvent, once chosen for a particular application, should be used throughout.

Cold trapping

Cold trapping is a second mode of reconcentration: where column temperature is low enough, vaporized components may condense into the liquid phase.

A general guideline is that components boiling at least 150°C above the column temperature will be reconcentrated by cold trapping at the head of the column. Components with lower boiling points are reconcentrated via the solvent effect.

Temperature programming

Multiple•rampoven temperature programming is advantageous: the oven is held at an appropriately **cool** temperature at injection to create an environment for component reconcentration.

It is then programmed upward rather quickly to release lighter components held by the solvent effect; then programmed higher yet, but at a lower rate, to release heavier components held by cold trapping.

Sample requirements

Injection volume is generally in the range 0.5 to 2 µl. Individual component concentrations must not be so great as to overload the column. Retention indices for components of interest should be greater than 600. The maximum amount of sample injected without column overload depends upon column bore, loading efficiency, and component polarity.

Inlet temperature

Because gas flow through the inlet insert during injection is reduced to that flowing through the column, sample residence time inside the insert is longer than with split sampling.

For this reason, relatively lower inlet temperatures may be used: a temperature in the range 150 to 200°C is adequate for most samples. Note that lower temperature also reduces volume required by vaporized sample, reducing possibility of sample **flash back**.

For samples with high boiling components (> C_{32}), a higher inlet temperature may be required.

A recommended procedure is to perform a series of analyses at increasingly higher inlet temperatures using components representative of those of interest, and analyzed using the conditions for later sample analyses. The optimum temperature is where maximum area counts are obtained, **and** there is no evidence of thermal degradation products.

Solvent purity

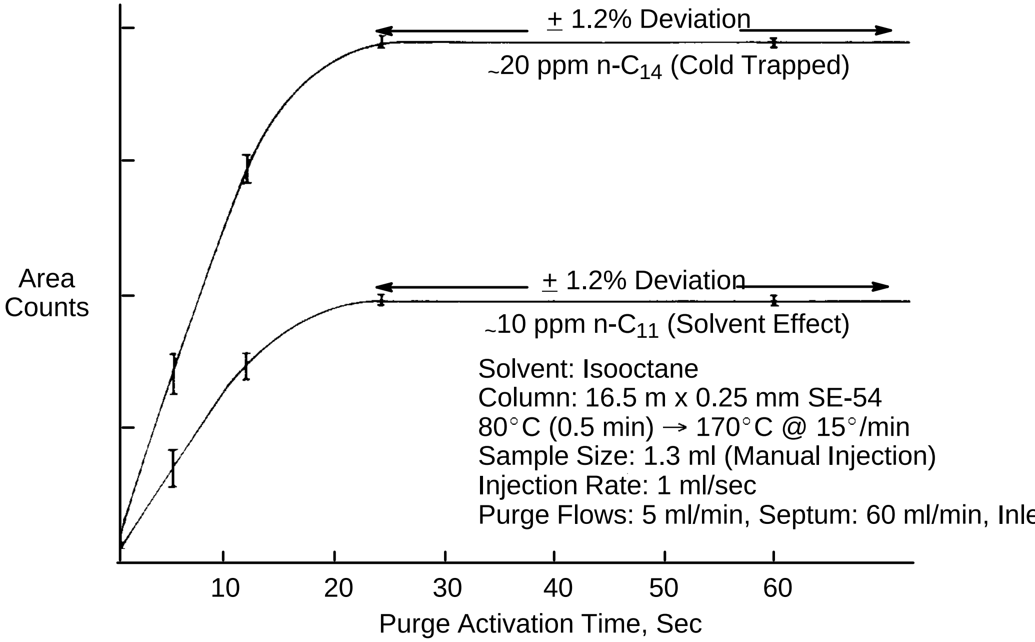
Reconcentration traps any solvent impurities at the head of the column, which may interfere with peaks of interest. A solvent blank run to check solvent purity is strongly recommended **before** the solvent is used to dilute samples for analysis.

Inlet insert purge

After injection, sufficient time is allowed for solvent and sample components to reconcentrate at the head of the column; then solvent vapor within the inlet insert is vented (purged).

The specific time delay after injection for insert purging to occur depends upon the components, solvent, and injection volume, flow rate through the inlet, and internal insert volume (approximately 1 ml); generally, a time within the range 0.3 to 2 minutes is reasonable.

Figure 6-10



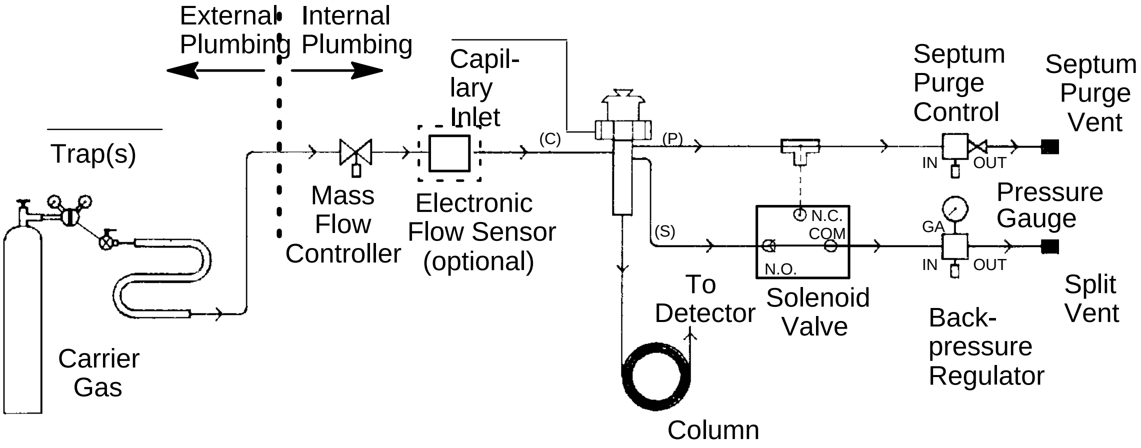
Effect of Inlet Purge Activation Time on Area Counts

Noting Figure 6•10,waiting too long does not increase component peak areas, but does increase interference by the solvent tail. Purging too early risks venting light components, not allowing sufficient time for heavier components to enter the column, and/or not having sufficient solvent enter the column to ensure good reconcentration.

A recommended procedure is to analyze a known standard using conditions identical to those to be used for later sample analyses. Ideally, the standard should be representative (in both components chosen and their respective concentrations) of unknown samples to be analyzed.

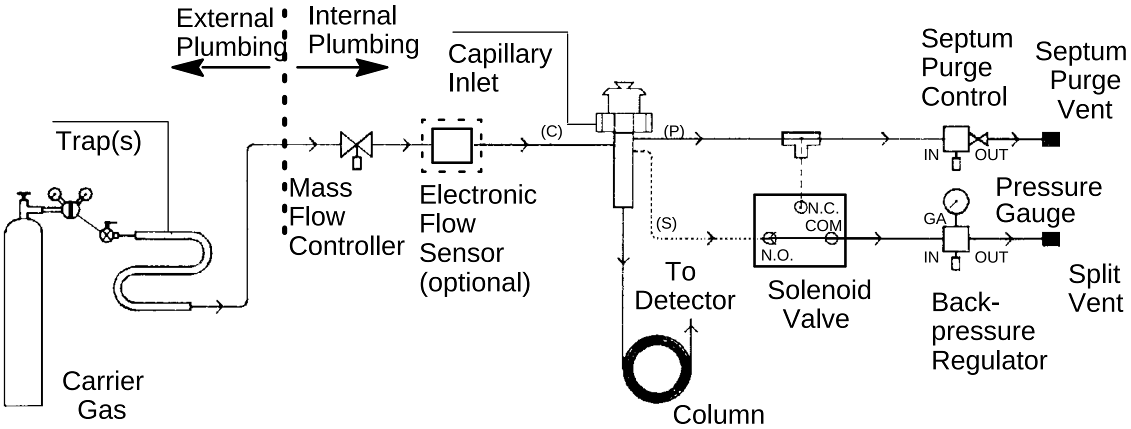
Perform a series of analyses with increasingly delayed insert purge times: the optimal time is where maximum area counts are obtained with least interference by the solvent.

Figure 6-11



Flow Diagram, Splitless Operation (inlet purge)

Figure 6-12



Flow Diagram, Splitless Operation (during injection)

Noting Figures 6•11 and 6•12,the splitless sampling process is as follows:

- **Before Injection:** Carrier gas flow enters through the mass flow controller, into the top of the inlet. A small fraction is split off to purge the septum and insert seal, then flows on to the purge vent.

  The remainder flows down the insert where it divides again: a fraction passes into the column while the rest flows around the bottom of the insert, up between the outside of the insert and the inlet body itself, and on to the split vent.

- **At Injection:** A solenoid valve (splitless solenoid valve) is actuated just prior to injection, to prevent carrier flow from exiting at the bottom of the insert. Flow through the insert now passes only through the column.

  The remainder flows through the top of the inlet to the split vent, via the solenoid valve and backpressure regulator.

- **After Injection:** At a predefined time after injection, the solenoid valve returns to its original state, restoring purge flow through the inlet insert.

In going from column flow + septum purge flow at injection (typically less than 5 ml/min), to total flow (column + split vent + septum purge) through the inlet (typically 50 to 100 ml/min), residual solvent vapor (1 to 5% of total injected solvent) is swept from the inlet.

Note that since carrier gas vents through the backpressure regulator, column head pressure (and, therefore, column flow rate) remains constant even when total flow through the insert is changed (injection versus insert purging).

Injection technique, split/splitless sampling

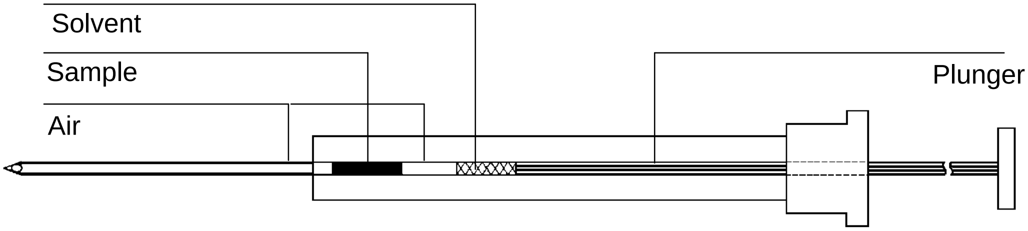
Proper injection technique is required for reproducible, quantitative data. The following steps should yield good results:

1. Rinse the syringe with solvent, completely filling and expelling the syringe several times.

2. Wipe excess solvent from the syringe needle.
3. Without introducing air, draw in excess sample.
4. Position the syringe plunger for the required injection volume. Wipe excess sample from the needle.
5. Draw in air until the sample/solvent is entirely within the syringe barrel. The sample is ready for injection.

This method results in the syringe filled as shown in Figure 6•13.

Figure 6-13



Properly Filled Syringe for Split or Splitless Sampling

Solvent following the sample (about 0.7 µl) helps to wash sample components from the syringe and needle bore.

For split sampling, with high gas velocity through the inlet, injection must be made in a continuous, rapid manner. Any lack of smooth motion may cause **multiple** injections.

For splitless sampling, with low gas velocity through the inlet, injection may be at a slower pace, but must also be done smoothly. Also, note that retention times depend upon amount injected, so total sample volume injected should be kept constant.

Note: In analyzing samples containing **heavy** components (e.g., C_{24} with a boiling point of 391°C), it may be necessary to leave the needle in the inlet for up to 10 seconds after injection. This reduces needle fractionation occurring with such components.

7

Detector Systems

Detector Systems

This chapter provides information for the five HP 5890 SERIES II (hereafter referred to as HP 5890) detector systems:

- Flame Ionization Detector (FID)
- Nitrogen•PhosphorusDetector (NPD)
- Electron Capture Detector (ECD)
- Thermal Conductivity Detector (TCD)
- Flame Photometric Detector (FPD)

Capillary makeup gas flow rate

Detectors are designed to operate best with a carrier flow rate of at least 20 ml/min, typical of packed column applications. Carrier flow rates less than about 10 ml/min (typically capillary applications) require capillary makeup gas to ensure a total flow rate (carrier + makeup) of at least 20 ml/min. The TCD is an exception, requiring a total flow rate of only 5 ml/min.

For the FID, NPD, and TCD, HP Series 530 μ capillary columns may be used without capillary makeup gas, as long as carrier flow rate is between 10 and 20 ml/min. Some loss of detector sensitivity may occur at lower flow rates.

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

Makeup gas is added directly to H_2 for an FID or NPD within the detector flow manifold; for an ECD or TCD, it is added into the column effluent stream via a capillary makeup gas adapter fitted into the detector column inlet.

Supply pressure for capillary makeup gas should be set to about 276 kPa (40 psi).

FID and NPD jets

Depending upon the column type to be used, and/or analyses to be performed, exchanging the jet in an FID or NPD may be necessary. Table 7•1lists available jets.

Note: If switching from packed column operation to capillary operation, the jet for capillary use must be installed **prior** to column installation.

Table 7-1. Available FID / NPD Jets

Part No.	Jet Tip ID (inch)*	Use
18789-80070	0.030	Packed Column Only (FID only: Simulated Distillation, TCD-to-FIS series operation.)
18710-20119	0.018	Packed Column (Standard, FID and NPD)
19244-80560	0.011	Capillary Column (FID and NPD) (FID: high sensitivity, packed column)

* Measured at the jet tip.

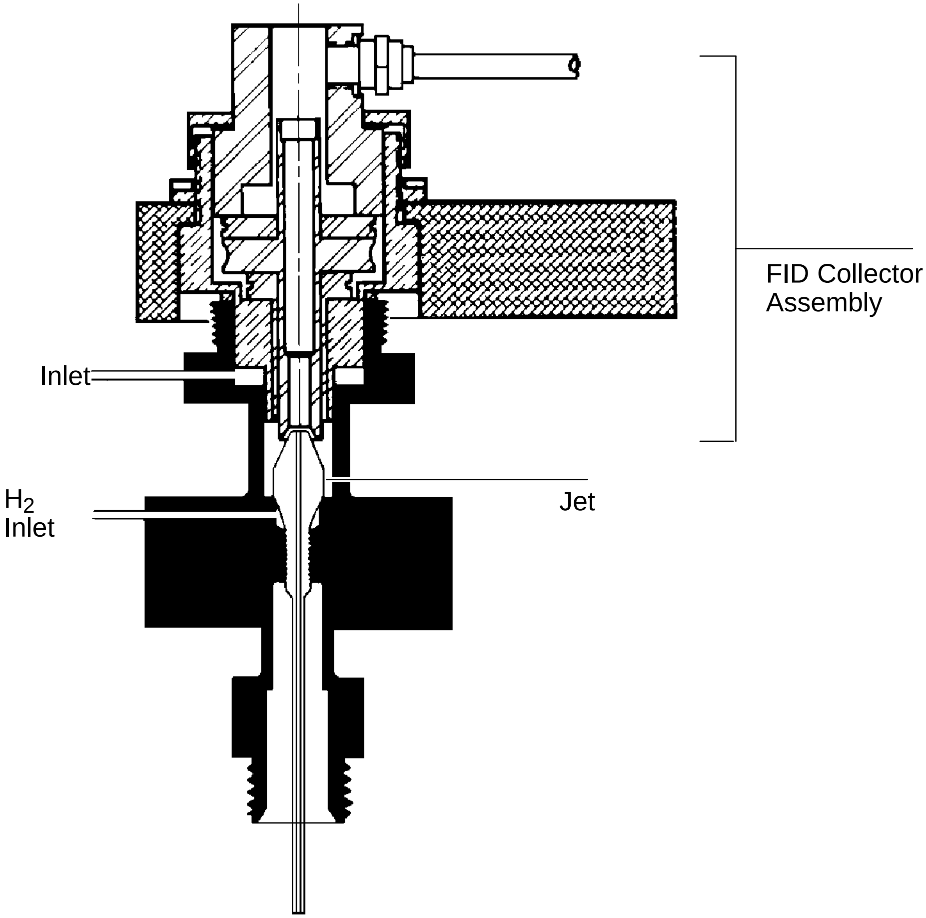
For an FID, the 0.011•inchjet maximizes detector sensitivity and **must** be used with capillary columns; if used with packed columns, flame•out and clogging problems may occur.

For an NPD, exchanging jets is less critical to detector sensitivity: the 0.011•or 0.018•inchjet may be used with either packed or capillary columns. The 0.011•inchjet **must** be used with capillary columns.

In packed column applications, the 0.011•inchjet may be tried first. The 0.018•inchjet may then be exchanged if problems are encountered. See Chapter 8**,** *Preventive Maintenance,* for specific details regarding jet exchange.

Flame ionization detector (FID)

Figure 7-1



Flame Ionization Detector (FID)

The flame ionization detector (FID) responds to compounds that produce ions when burned in a H_2•airflame. These include all organic compounds, although a few (e.g., formic acid, acetaldehyde) exhibit poor sensitivity.

Compounds producing little or no response include:

Rare gases
Nitrogen Oxides
Silicon Halides
H_2O
NH_3
N_2
CO
CO_2
CS_2
O_2
N_2
*CCl_4

---

* Measured at the jet tip.

This selectivity can be advantageous: for example, H_2O or CS_2, used as solvent, do not produce large solvent peaks.

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; it is directly proportional to sensitivity of the FID toward the given compound.

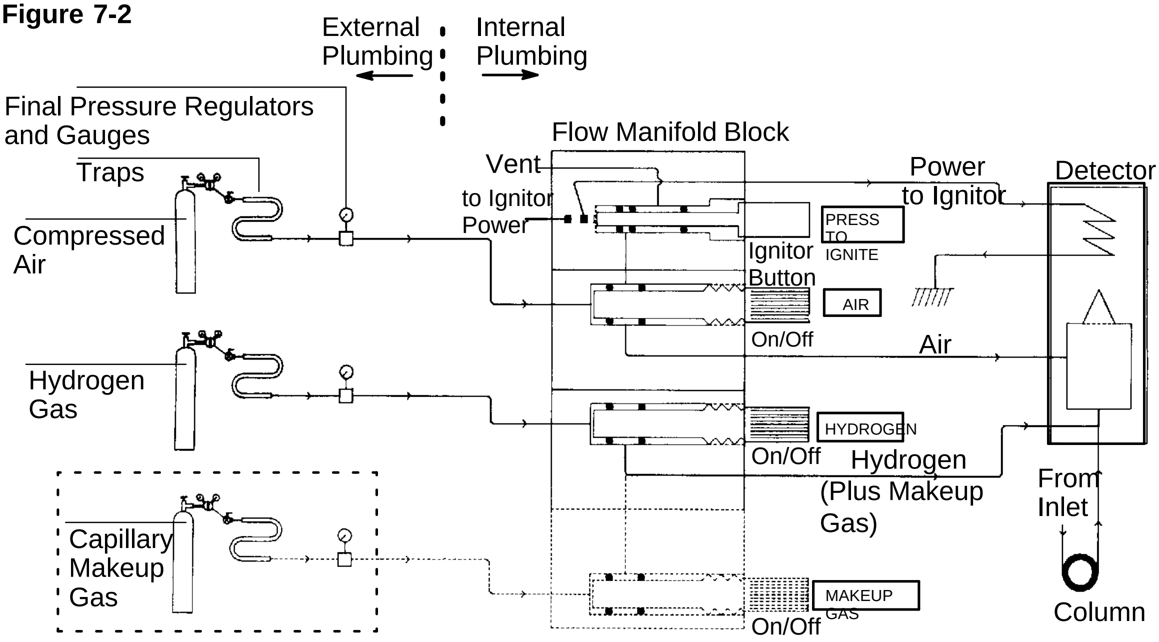
In general, where sample components of interest are in high concentration, increased air flow may be necessary (up to 650 ml/min). Where components of interest are in low concentration, reduced air flow rates are acceptable (375 to 425 ml/min).

For maximum sensitivity, it is recommended that a standard sample be made containing components of interest in concentrations expected. By experimenting with different carrier, air, and H_2 flow rates, the standard is used to determine flow rates giving maximum response.

FID flameout problems

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

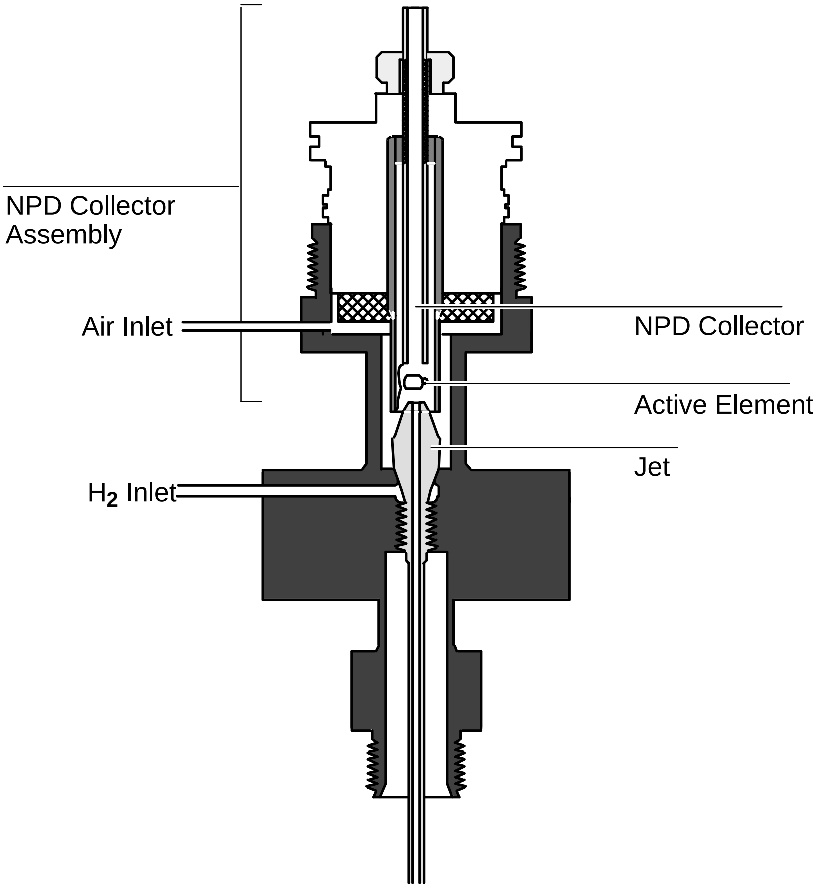
Figure 7-2



Flow Diagram, Flame Ionization Detector (FID)

Nitrogen-phosphorus detector (NPD)

Figure 7-3



Nitrogen-Phosphorus Detector (NPD)

The nitrogen•phosphorusdetector 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. In the presence of this thermionic source, nitrogen•and phosphorus•containing organic molecules are efficiently ionized. Ions are collected, and the resulting current is measured.

H_2 and air are required, but at flows significantly less than those for an FID. Normal FID•typeionizations are therefore minimal, so response to compounds not containing nitrogen or phosphorus is reduced. Thus, the detector is both sensitive to and selective toward only compounds containing nitrogen and/or phosphorus.

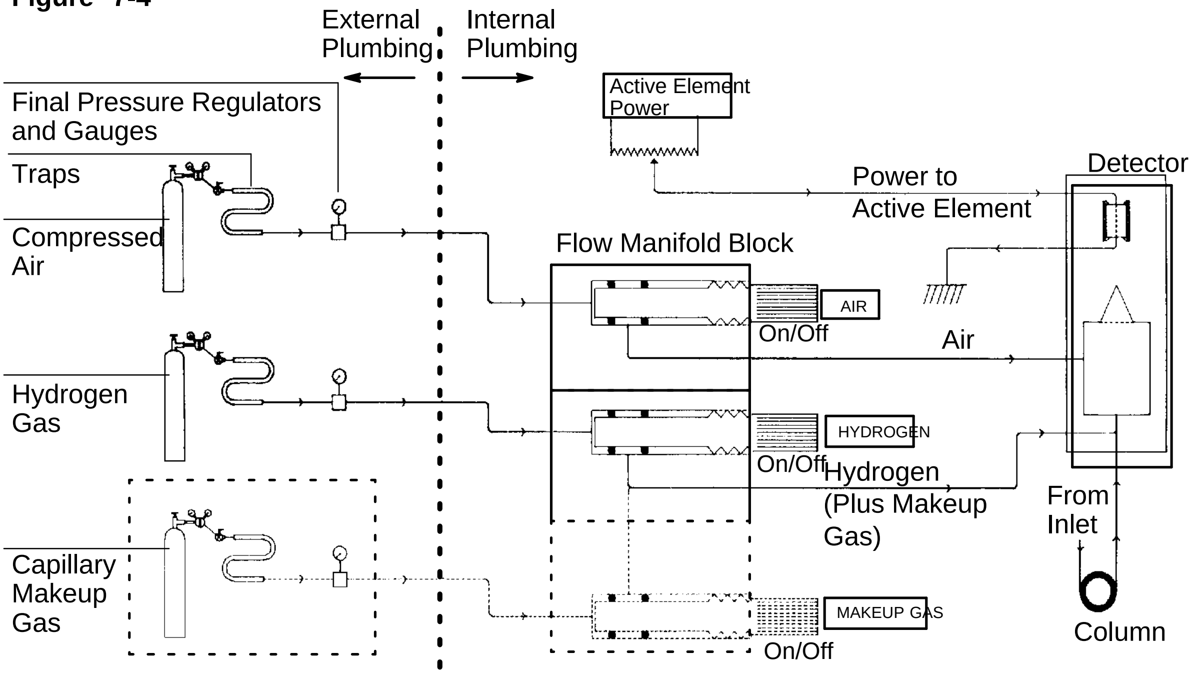
The electrical power for heating the active element is supplied through a toroidal transformer located inside the NPD detector cover. The toroidal transformer secondary winding is connected directly to the collector/active element assembly. The electrical heating current passes directly through the small 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 is dependent 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. Some of the important variables and their effects are listed below.

1. Increasing detector temperature. This increases the active element temperature and the response.
2. Increasing electrical power to the active element. This increases active element temperature and increases the response.
3. Increasing hydrogen flow. This increases the active element temperature as well as increasing the size of the active zone around the active element; both effects will result in increased response.
4. Increasing air flow to the detector. Normally this cools the active element slightly and decreases the response. (The overall effect is much less than the hydrogen flow effects.) Increasing the air flow also decreases the residence time of a given peak in the active zone of the active element and decreases response.
5. Increasing the carrier gas flow. This cools the active zone slightly, decreasing response. This also decreases the residence time of a component in the active zone and decreases response.

Other gas flow effects of too high flow rates of the hydrogen may allow a true flame to exist around the active element. This would overheat the active element severely and destroy the specific response. Too low flow rates of air tend to quench the background response of the active element, and this results in a re•equilibrationtime that is too long to establish proper background response (negative solvent peaks **killing** the active element).

Figure 7-4



Flow Diagram, Nitrogen-Phosphorus Detector (NPD)

Performance considerations

Contamination

Very little contamination can create serious NPD problems. Common sources include:

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

Contamination in the NPD can be either a positive type or a negative type of contamination. A positive type of contamination is one that gives a more positive offset than would normally result with a clean system. When this happens there is a tendency to try to operate the detector with too little power to the active element, with the final result that the detector does not appear as sensitive as desirable (because the temperature of the active element is actually less than normal).

If the contamination is a negative type of contamination, then there is a tendency to **quench** the reactions that give rise to the specific response desired. This **quenching** can be only partial, resulting in less sensitivity than expected, or at very high contamination levels, a complete quenching of all signals from the detector. This latter type of quenching is (usually) only seen on sample peaks where the apex of the peak actually goes back down toward the baseline.

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%.

Active element lifetime

Lifetime of the active element is reduced by the silicon dioxide coating, described above, and by irreversible loss of rubidium salt.

Rubidium loss is caused by overheating the active element, particularly if element power is on when gas flows, particularly carrier, are interrupted. The detector must be turned off or element power reduced to zero when changing columns, replacing septa, and/or replacing gas cylinders. Power to the element with gas flow off can destroy an element within a few minutes.

Humidity also affects element lifetime adversely: keep the detector warm (100 to 150°C) when not in use. A collector removed from an NPD for an extended period of time, or spare collectors, should be stored in a desiccator.

Element lifetime is extended by using the lowest element power possible, consistent with maintaining sufficient detector sensitivity and selectivity for the particular analyses.

Generally, sensitivity and selectivity toward nitrogen decreases first as the element ages. Phosphorus response is affected less.

It is good practice to have a spare collector available for replacement purposes. A recoating kit is available (Part No. 5080•8872)to rejuvenate the active element in an old collector.

Both detector baseline and sensitivity change with carrier flow rate due to change in temperature of the active element. This is the reason for the baseline drift in pressure•controlledinlet systems (capillary inlets) when temperature•programmingthe 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), i.e., total column flow change divided by makeup gas flow. Element power should be adjusted after any change in carrier flow rate.

Septa should be checked for leaks daily or replaced regularly. An upward baseline shift at injection is often due to a leaky septum.

Caution Turn the detector off to avoid damaging the active element before changing septa.

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 oven temperature to prevent contaminating the active element with column bleed.

To preserve the active element lifetime, it is good practice to 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 would be 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**.

Electron capture detector (ECD)

WARNING The effluent gas stream from the detector must be vented to a fume hood to prevent possible contamination of the laboratory with radioactive material.

Specific cleaning procedures are provided in Chapter 8, *Preventive Maintenance*.

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).

This 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•1798(D).

Owners of this detector must perform a radioactive leak test (wipe test) at least every six months. The procedure is described in Chapter 8, *Preventive Maintenance*.

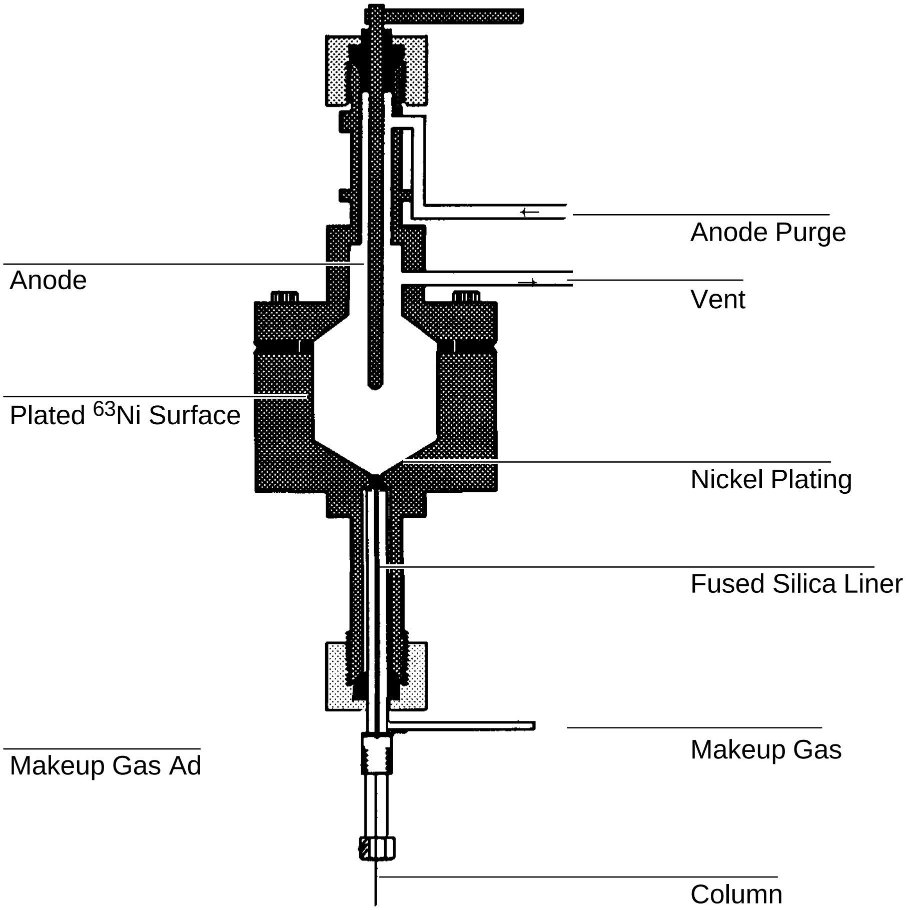
WARNING

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, the following must be done:

- After turning off main power and allowing the instrument to cool, cap ECD inlet and exhaust vent openings. Wear disposable plastic gloves and observe normal safety precautions.
- Return the cell for exchange, following directions included with the form General License Certification (HP Pub. No. 43•5954•7621).

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

Figure 7-5



Electron Capture Detector (ECD)

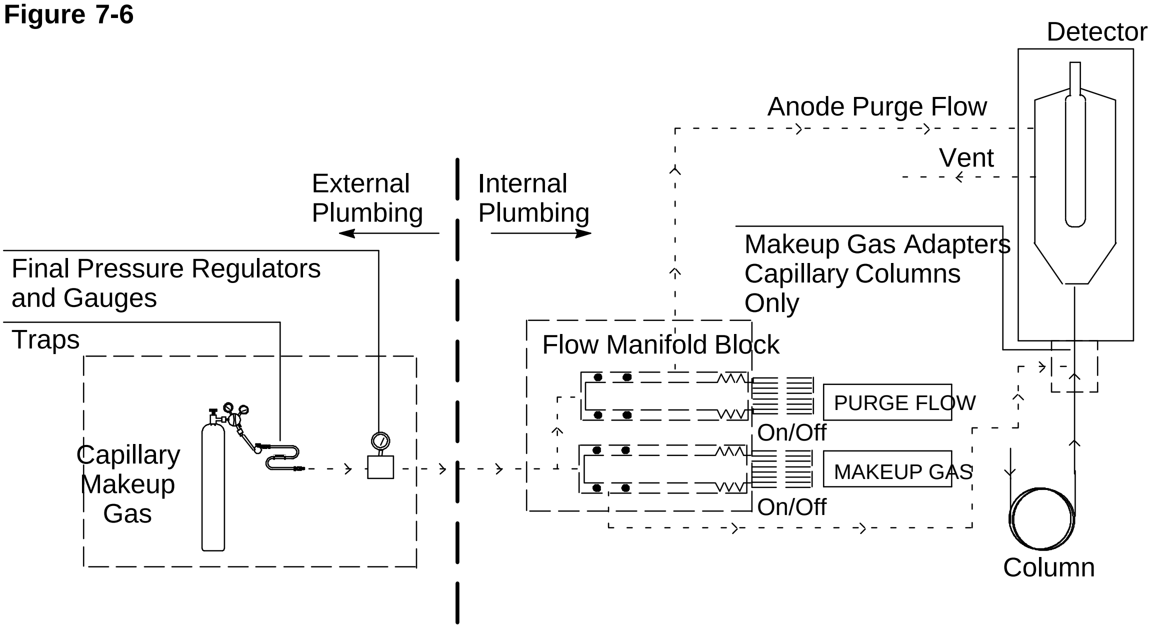
The electron capture detector (ECD) cell contains ^{63}Ni, a radioactive isotope emitting high•energyelectrons (β•particles).These undergo repeated collisions with carrier gas molecules, producing about 100 secondary electrons for each initial β•particle.

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

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

Therefore, pulse rate (frequency) rises when an electron•capturing compound is passing through the cell. The pulse rate is converted to a voltage, linearly related to amount of electron•capturingmaterial in the cell.

Figure 7-6



Flow Diagram, Electron Capture Detector (ECD)

Thus, the ECD responds to compounds having an affinity for electrons (for example, such halogenated materials as pesticides and related compounds). Table 7•2gives general indication of expected sensitivity to different classes of organic compounds.

Table 7-2. 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	100
Mono-Br, di-Cl and di-F compounds	1000
Anhydrides and tri-Cl compounds	10^4
Mono-I, di-Br and nitro compounds	10^5
Di-I, tri-Br, poly-Cl and poly-F compounds	10^6

These are only approximate figures; sensitivity varies widely within each group, depending upon structure of the material. For example, DDT (5 chlorine atoms/molecule) can be measured in the 1 to 10 picogram range.

The ECD is designed for use either with N_2 or with 5% CH_4 in Ar as carrier gas. A switch selects operation with either carrier gas; the ECD does not operate properly if the switch is incorrectly set. See Chapter 5 of the *Operating Manual.*

Because of its high sensitivity, the ECD should never be used without traps (moisture, chemical), should be in good condition, and should be installed in the carrier gas supply line. For capillary systems, this must also be done for the makeup gas supply.

An ECD is normally used to detect compounds that are reactive enough to interact with metal columns. Therefore, only 1/4•inchpacked glass, or fused silica, or glass capillary columns are recommended with this detector.

Considerations for packed column operation

Either N_2 or Ar containing 5 or 10% 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.

The carrier gas must be dry and O_2•free.Moisture and O_2 traps are strongly recommended for highest sensitivity. Because plastic tubing is permeable to many gases, use copper tubing for all connections.

Total flow into the detector must be at least 20 ml/min to prevent peak tailing.

Considerations for capillary column operation

H_2 carrier gas (with N_2 makeup gas) gives best column performance. Ar/CH_4 as makeup gas may also be used. For most purposes, 60 ml/min of makeup gas is satisfactory. For very fast runs this can be increased to 100 ml/min to sharpen peaks, but some sensitivity will be lost since the ECD is a concentration•dependentdetector.

H_2 or He carrier gas affords the best column performance with reduced retention times. Ar/CH_4 or N_2 makeup gas is used in the range of 30-60 ml/min. Since the ECD is a concentration dependent detector, reduced sensitivity is obtained at higher flow rates.

Moisture and O_2 traps for carrier gas are essential with capillary/ECD operation.

Temperature

Some compounds exhibit strong dependence of response to detector temperature. The effect may be either positive or negative. Different detector temperatures may be tried, always remaining above the oven temperature, to determine the effect on sensitivity. Generally a detector temperature between 250-300 °C is satisfactory for most applications.

Background level

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

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

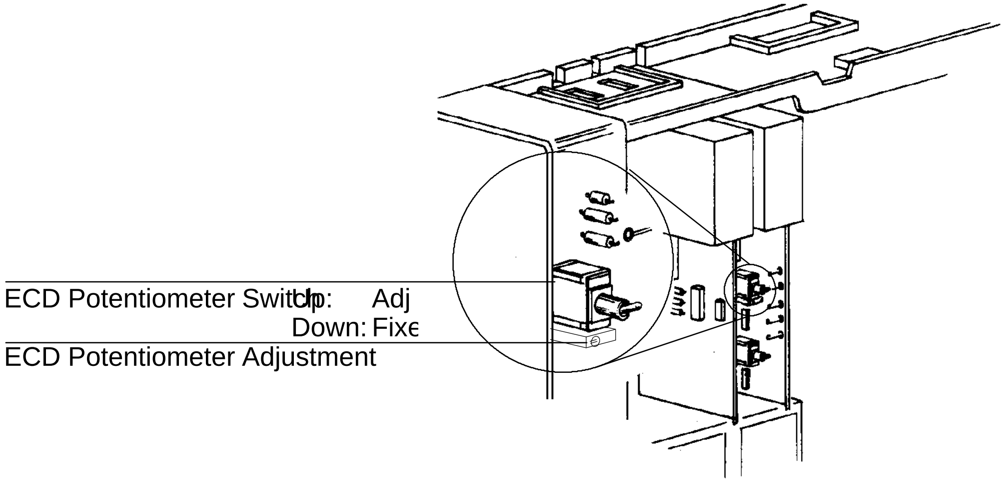
Prior to the run, enter the following:

DET A (or B) ON
SIG 1 (or SIG 2) A (or B)

Pressing SIG 1 (or SIG 2) again displays the signal source assigned to the particular output channel.

Ideally, the value displayed should be within the range 10 to 40 (100 to 400 Hz), indicating a clean system. For trace•levelanalysis, a reading of 20 to 60 is recommended. A large value of more than 50 (`500 Hz`) indicates a contaminated system and thermal cleaning should be performed (see Chapter 8, *Preventive Maintenance*).

A very clean system may produce a value below the low end of 10 (100 Hz). To correct this condition, an adjustment is made to the present potentiometer located on the ECD electronics board.

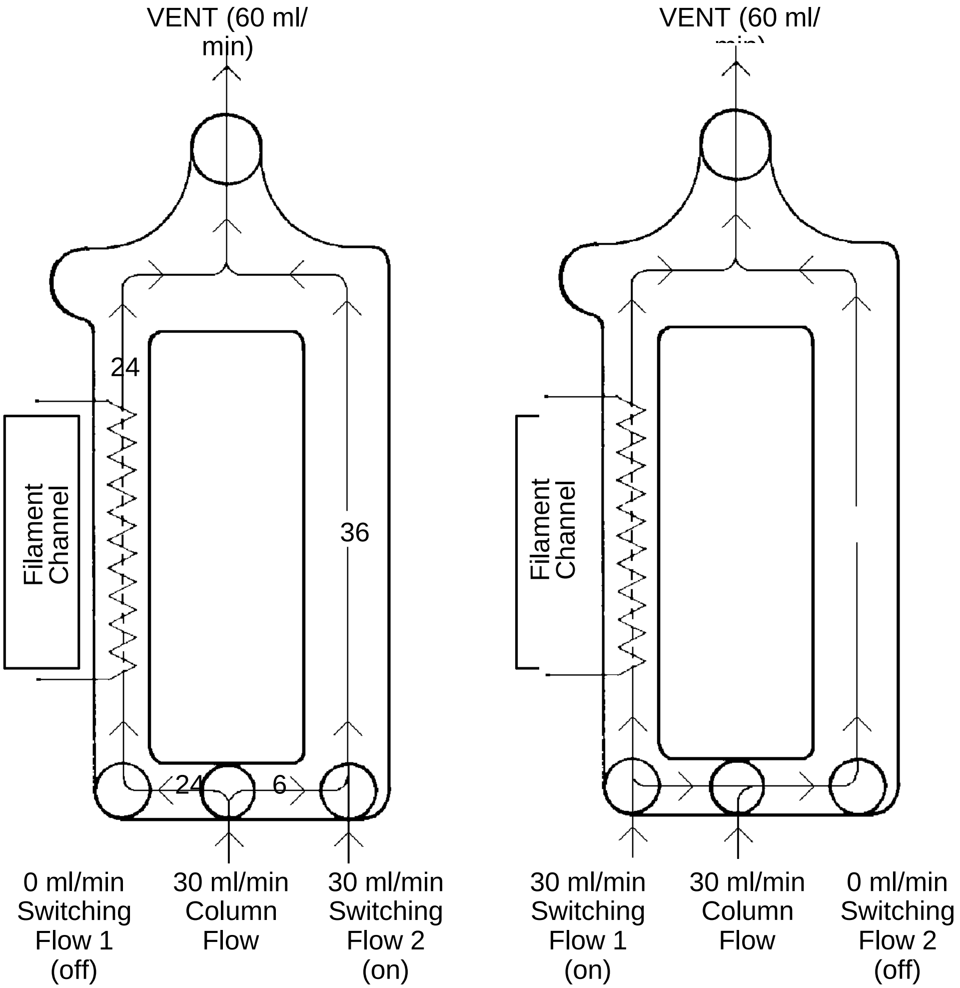


ECD Potentiometer Switch and Adjustment

1. Remove the right side of the panel.
2. Flip the switch up to the Adj. position.
3. Using a small screwdriver, turn the potentiometer clockwise until the background level is at or slightly above 20 (200 Hz, low reading for trace analysis).
4. Replace the right side of the panel.

Thermal conductivity detector (TCD)

Figure 7-7 **HP 5890 SERIES II TCD Cell**



Thermal Conductivity Detector (TCD)

The thermal conductivity detector (TCD) detects the difference in thermal conductivity between column effluent flow (carrier gas + sample components) and a reference flow of carrier gas alone; it produces voltage proportional to this difference. The voltage then becomes the output signal to the connected chart recording or integrating device.

The TCD uses a single filament to examine alternately relative thermal conductivities of reference versus column effluent gas streams every 200 msec. At this frequency, the detector is insensitive to thermal drift.

Factors which influence TCD response include the following:

- Temperature difference between the filament versus the surrounding detector block
- Flow rate ratio between carrier effluent versus reference gas streams
- Type of carrier/reference gas used

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/adjustments are made affecting gas flows through the detector.

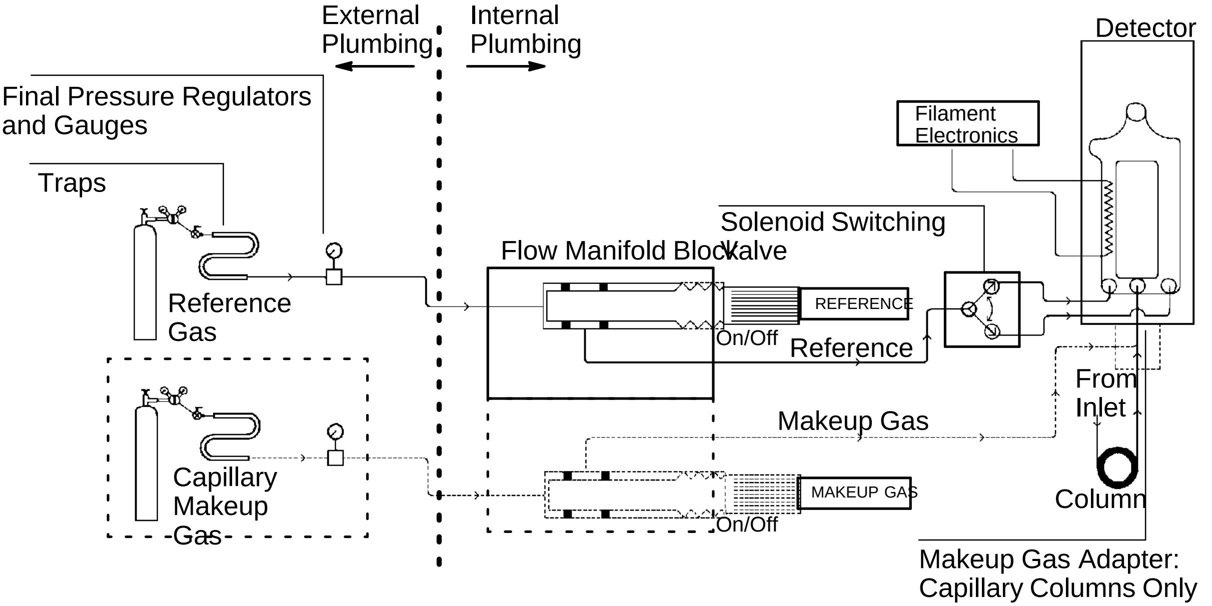
Likewise, exposure to O_2 can permanently damage the filament. Make sure the entire flow system associated with the TCD is leak•freeand that carrier/reference gas sources are uncontaminated before turning on the detector. Do not use Teflon tubing, either as column material or as gas supply lines, because it is permeable to O_2.

Since the TCD responds to any compound whose thermal conductivity is different from that of the carrier gas, H_2 and He are most commonly used as carrier gases, with H_2 giving somewhat greater sensitivity. However, H_2 forms explosive mixtures with air (O_2), and some components, particularly unsaturated compounds, may react with H_2. He produces almost as much sensitivity as H_2 and is free from problems of reactivity with sample components or the filament. N_2 or Ar may be used but give lower response for most materials; however, they are useful if H_2 or He is being analyzed.

Because of its exceptionally high thermal conductivity and chemical inertness, He is the recommended carrier gas: it gives large thermal conductivity differences with all compounds except H_2 (considerations necessary in H_2 analyses are discussed later). With He as carrier, the TCD exhibits universal response. For propane, the sensitivity limit is about 400 picograms/ml of He carrier gas.

The TCD exists in two configurations: either its exhaust vent tube exits at the top of the detector or the exhaust vent tube returns to the inside of the oven for connection to an FID or other device.

Figure 7-8



Flow Diagram, Thermal Conductivity Detector (TCD)

Optimizing performance

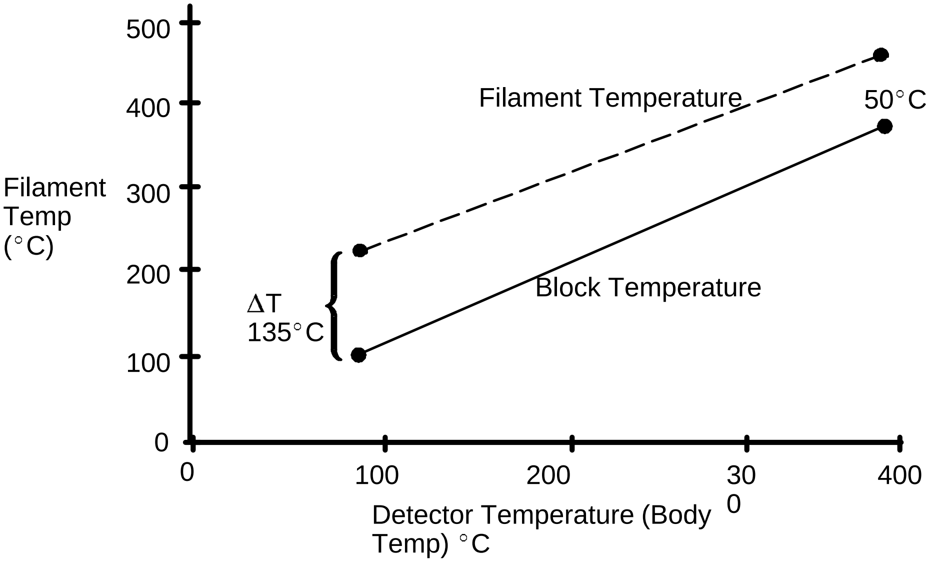
The following sections aid in choosing operating parameters to obtain optimal TCD performance.

Temperature

TCD sensitivity increases as the temperature difference between the detector filament (automatically set) and the surrounding detector body (chosen detector zone temperature) increases.

To minimize changes in detector sensitivity with different operating temperatures, filament temperature is maintained at a relatively constant difference above detector operating temperature. Filament resistance (proportional to temperature) is monitored, and power is adjusted as needed to maintain the filament at constant temperature.

Figure 7-9



Filament Temperature versus Detector Block Temperature

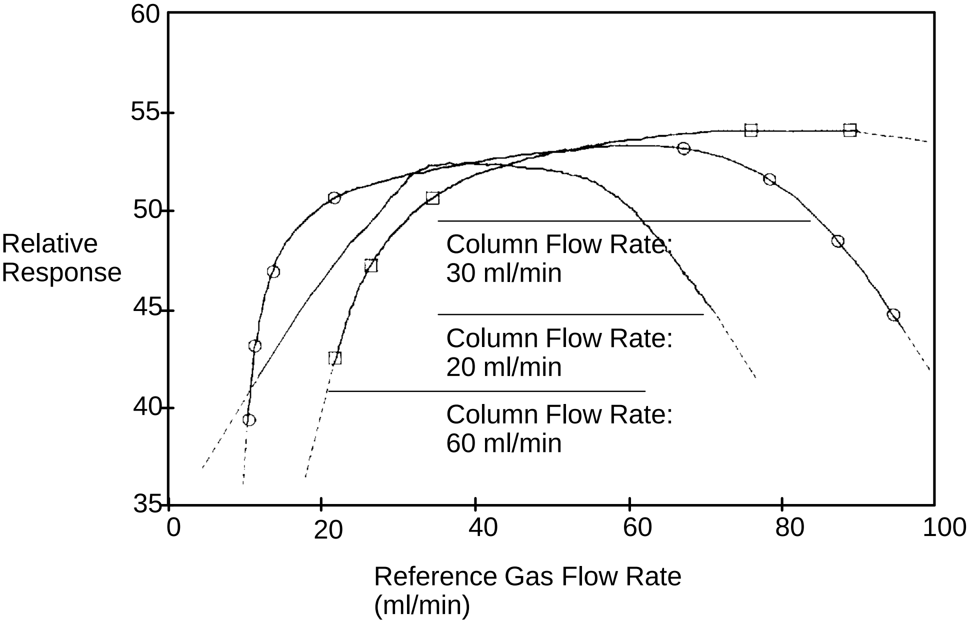
As Figure 7•9shows, however, the lower the detector zone temperature, the greater is the temperature difference between the filament versus the surrounding detector body temperature. Thus, for maximum sensitivity, the detector zone should be operated at the lowest temperature possible (limited by highest boiling components condensing inside the detector). Also, a second advantage is one of increased filament lifetime.

Gas flow rates

After the detector and instrument are allowed time for thermal stabilization (about 1/2•hourafter the oven and zones achieve desired setpoint values), two gas flow rates, column and reference gas, must be set independently.

After setting optimal carrier gas flow rate through the column, Figure 7•10may be used to determine the corresponding optimal reference flow rate.

Figure 7-10



TCD Response versus Reference Flow Rate

Note that TCD response becomes relatively flat (insensitive) to reference gas flow rates equal to, or somewhat greater than, flow rate through the column.

Analyzing for hydrogen, special considerations

Only H_2 has thermal conductivity greater than He. However, binary mixtures of small amounts of H_2 (< 20%) in He at moderate temperatures exhibit thermal conductivities less than either component alone.

Thus, depending on concentration and temperature, a H_2 peak may appear as positive, negative, a split peak, or as a W when using He as carrier. The phenomenon disappears at higher temperatures.

Note: Alternatively, N_2 or Ar may be used as carrier when analyzing for H_2; this eliminates problems inherent in using He as carrier, but detector sensitivity is substantially reduced toward components **other** than H_2.

To ensure normal peak shape in H_2 analyses with He as carrier, it is advisable to operate the detector at relatively high temperature (between 200 and 300°C).

A **specific** detector operating temperature is found by analyzing a known range of H_2 concentrations, increasing operating temperature until the H_2 peak exhibits normal shape and is always in the same direction (negative • relative to normal response to air or propane), **regardless** of concentration. This temperature also ensures high sensitivity and good linear dynamic range.

Since an H_2 peak is negative, detector polarity must be inverted at appropriate times so the peak appears positive to a connected integrator or chart recorder.

TCD-to-FID series connection

The following describes, for a TCD whose exhaust vent returns to the inside of the oven, connecting the TCD to an FID.

- If necessary (see NOTE below), exchange the standard FID jet for the 0.030•inchjet (Part No. 18789•80070). Information about jet exchange is available in Chapter 8**,** *Preventive Maintenance*.

Note: Use of the 0.030•inchjet is necessary only in cases where FID flame lighting problems occur due to increased gas flow rate through the FID, or TCD response is affected adversely due to the increased back pressure on the TCD.

Since the 0.030•inchjet reduces FID sensitivity, it is recommended that operation with the standard 0.018•inchjet be tried first, before deciding to switch to the 0.030•inchjet.

- Inside the oven, connect the TCD oven•returnexhaust vent tube to the base of the FID using the TCD•to•FIDjumper tube (Part No. 19302•80600) and standard 1/8•inchstainless steel swage•typenuts and ferrules.

  Ferrules should be preset, and two wrenches should be used in opposition in tightening the nut to prevent twisting the tube.

Note: Use of the TCD•to•FIDjumper tube is critical to achieve lowest possible detector background noise.

This completes series connection of an FID with a TCD.

Filament passivation

The TCD filament is a tungsten•rheniumalloy whose surface has been chemically passivated.

Passivation protects against damage due to O_2, but chemically active components such as acids and halogenated compounds may attack the filament. The immediate symptom is a permanent change in detector sensitivity due to change in filament resistance.

If possible, such offending materials should be avoided. If this is not possible, the filament may have to be replaced frequently.

Capillary column considerations

The TCD cell filament channel has an internal volume of about 3.5 µl. This small cell volume makes it suitable for use with capillary columns. In performing capillary analyses with the TCD, the following should be noted:

- Since a portion of the column passes through the TCD heated block and into the cell itself, zone temperature for the TCD should not be set greater than the maximum temperature allowed for the column. A higher zone temperature may cause column bleed.
- After setting column flow rate, makeup gas should be set so total flow rate (column + makeup) is 5 ml/min. Reference gas should be turned off while making this measurement.
    - Measure the column flow rate.
    - Open (counterclockwise) the On/Off valve for TCD makeup gas flow.
    - Use a small screwdriver to adjust the variable restrictor at the center of the On/Off valve as necessary to obtain the desired total flow rate.
- After makeup gas is adjusted, reference gas should be 1.5 times the total flow from the column plus makeup. Therefore, if column plus makeup flow is 30 ml/min, then reference flow = 45 ml/min. Also, with makeup, the column should be pushed all the way up into the detector and backed off approximately 1 mm. However, when not using makeup gas (when using Series 530 at a relatively high flow rate), then 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 and set it to 1 to 2 ml/min.

Flame photometric detector (FPD)

Optimizing FPD sensitivity and selectivity

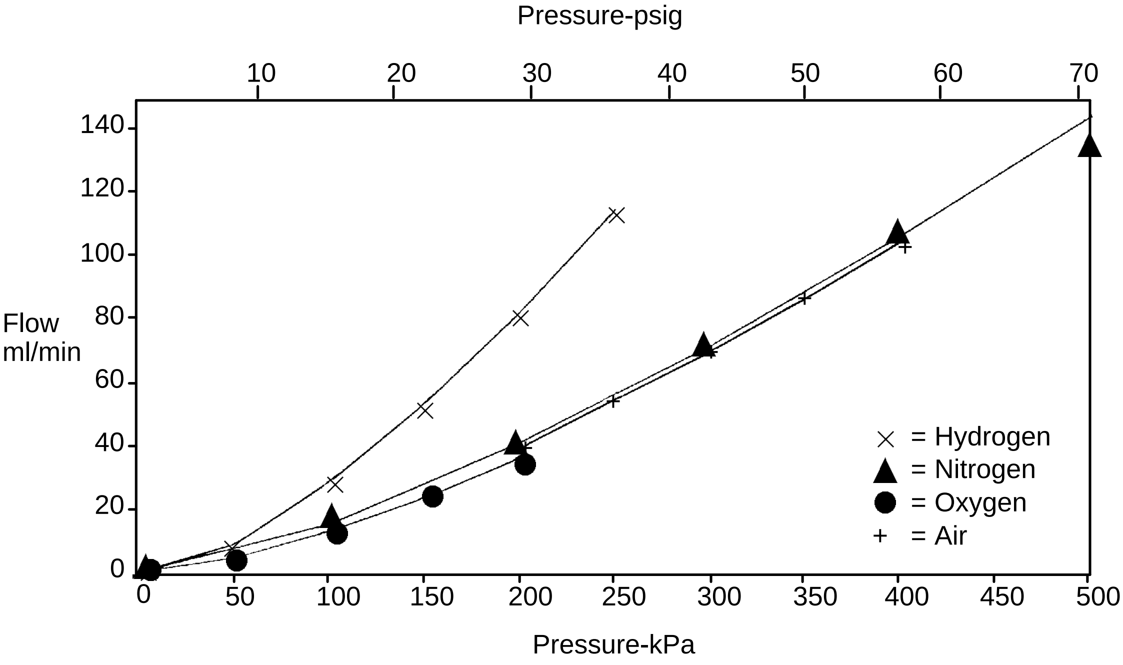
FPD sensitivity and selectivity are affected by several important parameters. These are listed below, with suggested ways to optimize for each application.

A. FPD Flow Rates. FPD flow rates are the most critical for optimizing either sensitivity or selectivity (these do not necessarily have the same optimal conditions). The most critical flow parameter is the hydrogen/air (or hydrogen/oxygen) ratio. Less critical are the auxiliary nitrogen/carrier combined flows.

A suggested way to set near•optimalflows is to begin with recommended flow rates located in the *HP 5890 Operating Manual*. Then vary each gas until a local maximum is reached. Optimize hydrogen first, then air (or oxygen), and lastly the auxiliary nitrogen flows. This may require a couple of iterations (tries). Adjusting these flows is most easily done by just varying the supply pressure regulator setting. Figure 7•11 plots approximate FPD flows versus supply pressure for all FPD gases.

Note: Take care that adjusting the supply gas pressure does not change some other flow supplied by the same regulator.

Figure 7-11



FPD Flows versus Supply Pressures

B. Detector Temperature. Detector heated zone temperature can have a significant effect on sensitivity. If analyzing thermally labile or very unstable compounds, a lower heated zone temperature may give the best results. If analyzing compounds with high boiling points, the detector temperature should be set at least 25°C but not > 300°C above the final oven temperature (but not greater than 300°C!).

C. Quenching. Quenching can result to some degree if sulfur or phosphorus peaks coelute with large hydrocarbon peaks. For many applications, this effect can be reduced by better chromatographic separation of peaks by varying the oven temperature program conditions and/or column selection. In cases where there is a large continuous hydrocarbon background, better selectivity of sulfur to carbon may be obtained by adjusting the hydrogen or air (oxygen) flow rate.

Flame ignition problems

Two common flame ignition problems are:

A loud **pop** results on ignition and the flame will not light or stay lit.

If a loud **pop** occurs on ignition, it is usually caused by an incorrect ignition sequence. The correct ignition sequence is:

1. Open the auxiliary Nitrogen Valve if required.
2. Open the Air/Oxygen Valve fully counterclockwise (CCW).
3. Press in and hold the Ignitor Valve.
4. Open the Hydrogen Valve fully counterclockwise (CCW). On doing this, there should be a slight **pop**.

Note: A loud **pop** is caused by opening the hydrogen valve **before** pressing the ignitor, thereby igniting a volume rich in hydrogen. This should not damage the detector but is unpleasant to hear. Always open the hydrogen valve **after** pressing the ignitor.

If the FPD flame won't light or stay lit, check/do the following:

1. Be sure there is a problem. Under some conditions, the flame lights so quietly that the **pop** is nearly inaudible. Also, especially in oxygen mode, the increase in signal offset on the LED display is usually only a few counts. Flame ignition is best verified by holding a mirror or shiny surface near the aluminum exhaust tube (with the rubber drip tube removed) and observing condensation (if the flame is lit).
2. If the flame doesn't light at all, be sure the glow plug circuit is working. This is most easily done by observing the LED display, which should go to greater than 65500 counts when the ignitor switch is pressed. If the LED display doesn't change with the ignitor switch pressed, either the circuit is open (check the pin connections at the detector flow manifold block, the lead connection on the glow plug, and the appropriate 5A fuse on the instrument's main circuit board).

3. Under some operating conditions, it is important to continue to hold the ignitor switch in for several seconds after opening the hydrogen valve fully counterclockwise.
4. Under some operating conditions, the flame may be more easily lit with the rubber drip tube removed. After lighting the flame, reinstall the drip tube and FPD cover assembly.
5. Under some conditions, the flame may be more easily lit with the detector temperature raised to 200°C or higher (but do not exceed the detector's maximum temperature of 300°C) and ignite the flame. After the flame is lit, set the detector to the required temperature.
6. If none of the above are sufficient to light the flame, try increasing the hydrogen supply pressure by 20-40%. After igniting the flame, reduce the hydrogen supply pressure to the initial value.
7. If the flame still won't light after trying the preceding steps, there may be a large leak in the system. This can result in measured flow rates being significantly different from actual flow rates, resulting in nonideal ignition conditions. Thoroughly leak•checkthe whole system.

8

Preventive Maintenance

Preventive Maintenance

This chapter includes maintenance, cleaning, and leak•testingHP 5890 SERIES II (hereafter referred to as HP 5890) inlet and detector systems.

Conditioning columns

Columns may contain contaminants; conditioning drives off unwanted volatiles, making the column fit for analytical use.

New packed columns should be conditioned since 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.

Conditioning is not a serious problem with capillary columns since there is little stationary phase. For the same reason, however, conditioning must be gentle not to drive stationary phase from the column.

The following are general guidelines for conditioning:

1. **Switch off detectors!** Shut off support gases, if any, to detectors (particularly H_2!).
2. a. If the column to be conditioned is already installed, disconnect its detector end.

   b. If the column to be conditioned is not already installed, connect one end to an available inlet. **Do not** connect the remaining end to a detector!

   For a split/splitless (or split•only)capillary inlet, if a capillary column is to be conditioned, install the proper insert and attach the column in the normal manner, making certain about 7.5 mm of column extends above (in front of) the column nut (∼25 mm from back of nut). Adjust the septum purge flow rate to no more than 6 ml/min.

   c. Cap inlet fittings into detector(s) to prevent entry of air and/or contaminants.

3. Establish a stable flow of carrier gas through the column. He is preferred; however, N_2 is adequate for conditioning packed columns. Do not use H_2 because it vents into the column oven during conditioning.
   a. For 1/8•and 1/4•inchcolumns (with 2 mm id), a flow rate of about 30 ml/min is sufficient; for 1/4•inchcolumns (with 4 mm id), a flow rate of about 50 ml/min is adequate.
   b. For a rigid glass capillary column (with 0.25 mm id), establish a head pressure (in psi) equal to about half the column length (in meters). For example, a 50 m column should have a head pressure of about 25 psi (172 kPa).
   c. For a fused silica capillary column, establish a column head pressure (in psi) equal to column length (in meters).
4. Set oven temperature to 100°C for about an hour. Then slowly raise oven temperature to the conditioning temperature for the column (never greater than the maximum temperature limit for the column; 30°C less than the maximum is usually sufficient).

Caution Overheating may shorten column lifetime.

Conditioning may continue overnight for packed columns and can be much less for capillary columns.

5. If the conditioned column is not to be used immediately, remove it from the oven and cap its ends to prevent entry of air, moisture, and/or contaminants.

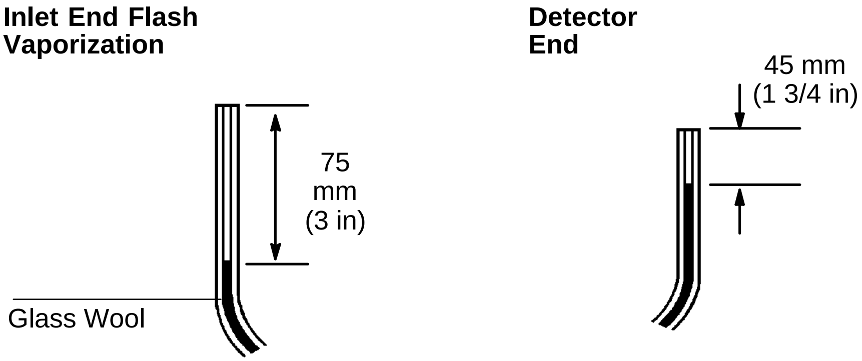
(Re)Packing columns

In packing columns (particularly 1/4•inchglass columns), one must consider the type of packing, column bore, and type (metal or glass), the method of sample introduction (flash vaporization or on•column),inlet or detector base requirements.

The method of sample introduction and/or the inlet/detector configuration determines the distance from the column end at which packing should start. Only general guidelines can be given here.

1. For FIDs and NPDs, at the detector end of the column, make sure neither packing nor a glass wool plug retaining the packing comes in contact with the bottom of the detector jet.
2. For 1/8•inchcolumns, where flash volatilization is used, packing should be as close to the inlet end of the column as feasible, leaving room for a glass wool plug to retain packing material.

Figure 8-1



Packing Limits for 1/4-inch Glass Columns

3. For 1/4•inchglass columns, since the inlet end serves as a liner, room must be provided to ensure flash volatilization and to ensure the syringe needle does not directly contact the glass wool plug and/or packing material. See Figure 8•1for recommended packing limits.

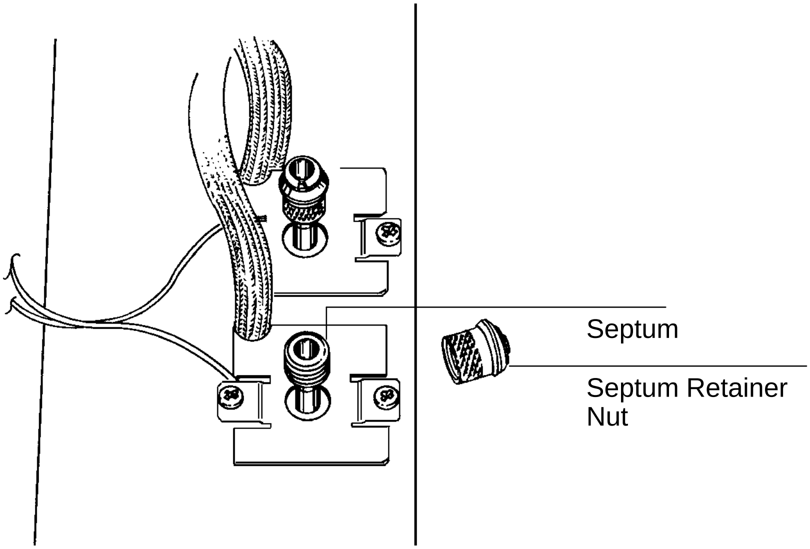
Packed column inlet

Changing septa

Septum lifetime is dependent upon frequency of use and upon needle quality; burrs, sharp edges, rough surfaces, or a blunt end on the needle decreases septum lifetime.

A leaking septum is evidenced by longer retention times, loss of response, and/or loss of column head pressure as well as degradation in detector signal quality (the signal becoming increasingly noisy). Where the instrument is used on a regular basis, daily septum replacement is recommended.

Figure 8-2



Packed Column Inlet, Replacing the Septum

Caution Turn off the detector (particularly a TCD or NPD!) before interrupting column flow.

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

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

Turn off carrier flow and decrease head pressure to zero. Remove the septum retainer nut and old septum. Insert a new septum. Replace the septum retainer nut, tightening it to finger•tightness(as tight as possible by hand without using any kind of tool). Do not overtighten; the retainer nut is spring•loadedto provide sufficient pressure for sealing. Restore carrier flow.

Insert/liner care

Cleaning inlet inserts and/or liners is discussed under *Liner and/or insert care*, later in this chapter.

Leaks

Leaks in the gas plumbing system can affect chromatographic results dramatically. The following procedure checks the flow system up to but not including the column connection. If this portion of the system proves to be leak free, refer to procedures later in this chapter to leak•checkthe detector.

1. Turn off detectors. As necessary, set oven and heated zone temperatures to ambient and allow zones to cool. Turn off gas supplies at their sources.
2. Remove any installed column and cap the inlet at its column fitting.
3. Turn on gas to the inlet at its source and adjust its supply pressure to 344 kPa (50 psi).

4. Fully open the mass flow controller counterclockwise and wait 1 to 2 minutes to ensure equilibrium.
5. Turn off gas to the inlet at its source.
6. Wait 10 minutes while observing carrier source pressure. If it drops less than 7 to 14 kPa (1 to 2 psi), the system (through the inlet column fitting) is considered leak free.

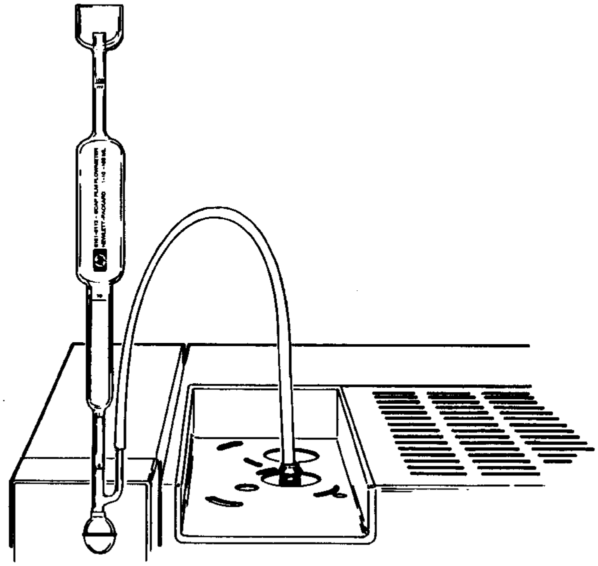
Evidence of leakage requires the inlet system be leak•checked. Repressurize the system as necessary, and

- Use a suitable leak•detectionfluid to check for leakage fittings. Correct or replace leaking fittings as needed.

Caution Leak•detectionfluids often leave contaminating residues; after each application, the area checked should be rinsed with CH_3OH (methanol) and allowed to dry.

- After verifying fittings are leak•free,there are two locations at the inlet itself where leakage may occur: at the septum, and at the column connection.
    - The column connection is checked using a leak•detectionfluid.
    - The septum is checked with a bubble flow meter by pressing its rubber inlet tube tightly onto the needle entrance in the septum retainer nut or simply replacing the septum.

Figure 8-3



Packed Column Inlet, Leak-Checking the Septum

Cleaning

Turn off the heated zone for the inlet and allow it to cool.

Remove the septum retainer nut and septum; remove also the column and inlet liner. Using a suitable light source, illuminate the inside of the inlet from inside the oven while looking through the inlet from the top. If there is evidence of contamination or deposits, the inlet should be cleaned.

Use a swab and an appropriate solvent to scrub interior walls until deposits are removed. A suitable wire may be used to dislodge solid particles carefully. Dry thoroughly with compressed, filtered, dry air or N_2 before reassembling.

WARNING To avoid possible eye injury, wear eye protection when using compressed gas.

Split/splitless capillary inlets

Changing septa

For a conventional disk•typeseptum, lifetime is dependent upon needle quality; needles should be sharply pointed and free of burrs or rough surfaces.

Choice of septum material is less critical than with a packed column inlet since the septum is continually purged. Thus, septa can be chosen primarily for durability. Septa, 11 mm (Part No. 5181•1263,package of 25) are recommended.

Note that regardless of the material, septa must be clean and free of particulate matter.

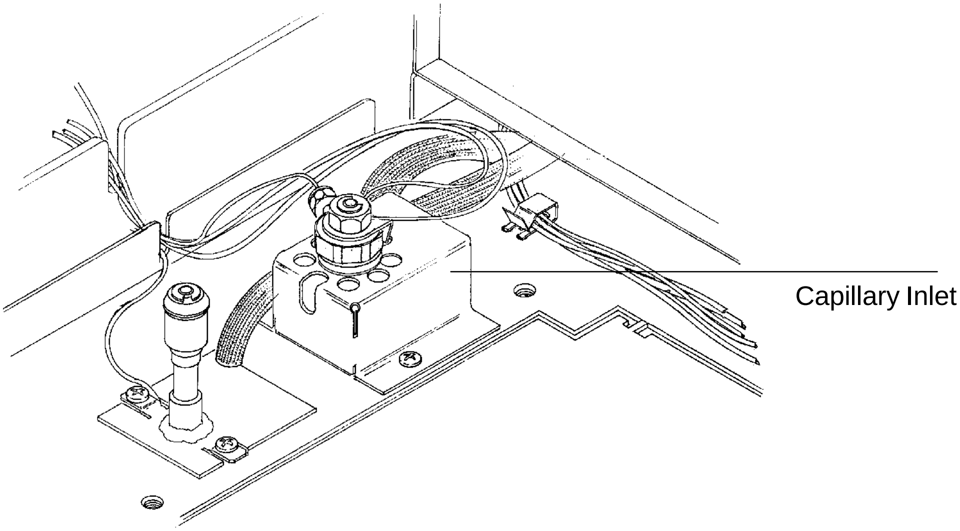
WARNING Exercise care! The oven and/or inlet or detector fittings may be hot enough to cause burns.

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.

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

1. Loosen and remove the septum retainer nut. Remove and discard the old septum, found either in the top of the inlet or inside the septum retainer nut.

Figure 8-4



Septum Replacement, Split/Splitless and Split-Only Capillary Inlet

2. The new septum is placed in the top of the inlet base. Make sure that sealing surfaces at the top of the inlet and inside the retainer nut are clean (no particulate matter).
3. Replace the septum retainer nut, tightening it to finger•tightness(as tight as possible by hand without using any kind of tool). Do not overtighten; the retainer nut is spring•loadedto provide sufficient pressure for sealing.

Insert care

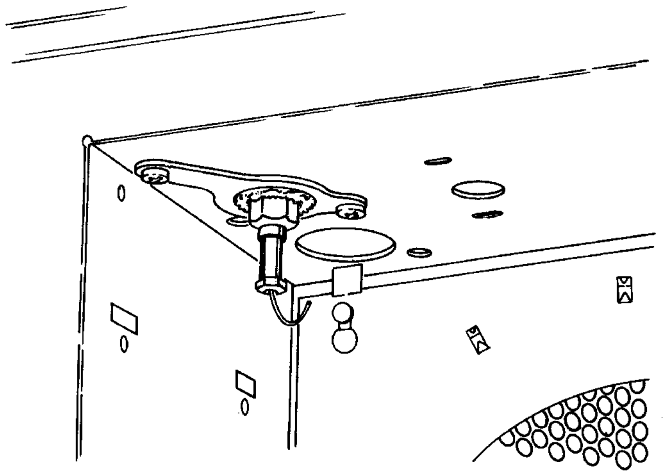
Cleaning inlet inserts is discussed under *Liner and/or insert care*, later in this chapter.

Leaks

For proper inlet operation, it is essential the entire system be leak•tight. The following procedure should be performed in initial checkout, or any time a leak is suspected.

1. **Switch off detector!**
2. Install an inlet plug (a paper clip or similar•gaugewire) in the same manner as a capillary column.

Figure 8-5



Capillary Inlet Plug Installed for Leak Test

3. Adjust the split•flowflow controller to about 60 ml/min.
4. Adjust or set the column head pressure to obtain a **column** pressure of 138 kPa (20 psi).

[gold] [INJ B PRES] [2] [0] [ENTER] *Set inlet B pressure to 20 psi.*

5. Shut off septum purge or cap the septum purge vent with a suitable swagelok fitting. Verify that it is shut off with a soap film (bubble) flow meter.

6. Turn off flow to the inlet by turning off carrier gas at the flow controller (**fully** clockwise, turning it **only** until it **bottoms**, and then **no** further).
7. Adjust the back pressure regulator **clockwise**, an additional 1/4•turn or set the electronic pressure control to 145 kPa (21 psi) and observe **column** pressure at the gauge for about ten minutes.

If the system is leak•free,pressure will remain between 131 and 138 kPa (19 to 20 psi); if it drops, a leak exists. If there is leakage, turn on the carrier gas supply and

- Use a suitable leak•detectionfluid to check appropriate plumbing connections behind the flow panel and onto the supply itself. Repair or replace fittings and components as necessary.

Caution Leak•detectionfluids often leave contaminating residues: After each application, the area checked should be rinsed with CH_3OH (methanol) and allowed to dry.

- Check the inlet itself at three possible locations:
  - The septum.
  - The column connection.
  - The seal (O•ring)around the insert retainer nut.

Note: The following checks may be done at any time during normal operation without either disconnecting the column or sealing the inlet. Simply pressurize the system and check for overall leak•down.

- Check for septum leakage by using a bubble flow meter: press its inlet tube onto the top of the inlet needle guide (septum retainer nut). Making sure the tube is flush with the surface of the inlet, check the bubble meter for evidence of leakage. Replace the septum if leakage is observed.

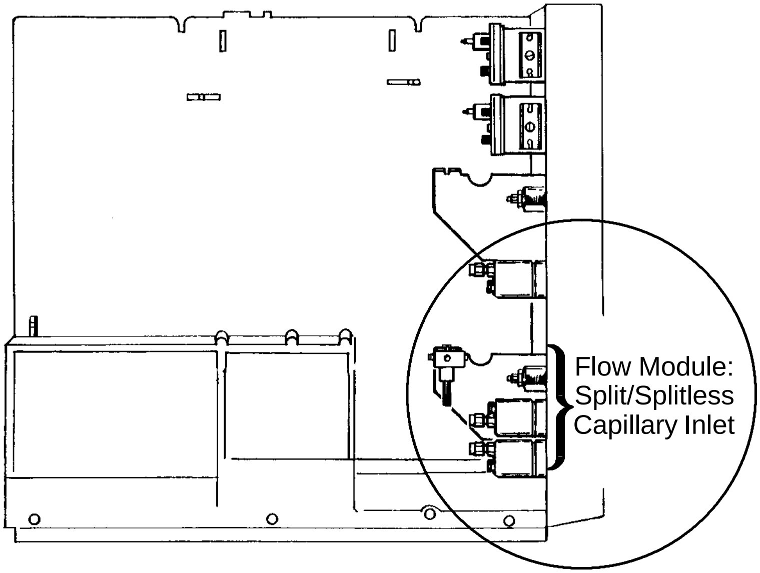
- Use leak detection fluid to check for leakage at the column nut. If leakage is observed, try tightening the nut first. If leakage continues, replace the ferrule.

  Note that if the inlet is hot, leak detection fluid may boil, giving false indication of a leak.

- If the septum and column nut prove to be leak•free,replace the seal (O•ring)on the inlet insert.

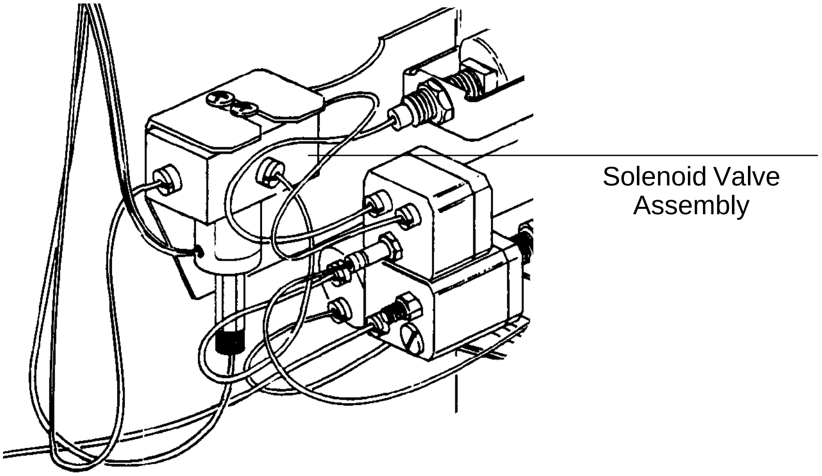
  Repressurize the system and check again for overall system leak•down.

Figure 8-6



Location of the Split/Splitless Capillary Inlet Solenoid Valve

Figure 8-7



Solenoid Valve, Split/Splitless Capillary Inlet

Cleaning

Turn off the heated zone for the inlet and allow it to cool.

Remove septum retainer nut, septum, insert retainer nut, and inlet insert; also remove the column. Using a suitable light source, illuminate the inside of the inlet. If there is evidence of contamination or deposits, the inlet should be cleaned.

Use a swab and an appropriate solvent to scrub interior walls until deposits are removed. A suitable wire may be used to dislodge solid particles carefully. Dry thoroughly with compressed, filtered, dry air or N_2 before reassembling. Vent lines can be removed for cleaning or replaced as necessary.

Compressed, dry air or N_2 may also be used to blow out any loose contaminating particles.

WARNING To avoid possible eye injury, wear eye protection when using compressed gas.

Liner and/or insert care

Regardless of the inlet system, inlet inserts and/or liners must be kept clean for optimum performance, particularly their interiors from which contamination may enter the column and/or interact with sample components.

Note: Excessive contamination anywhere on an insert or liner should be avoided, particularly its interior.

Ideally, clean replacement liners and/or inserts should be available for quick exchange when necessary.

Liners and/or inserts are treated differently, depending upon whether they are of glass or metal.

Glass inserts

- If a split insert (for a split/splitless, or split•only capillary inlet system) is to be cleaned, remove its packing.
- Because contamination is often in the form of carbonaceous materials (insoluble in organic solvents), immersion in concentrated chromic acid (for up to 24 hours) is recommended as the first cleaning step.

WARNING Concentrated chromic acid is highly toxic and corrosive. Observe proper safety precautions.

- After chromic acid, inserts are rinsed in distilled H_2O, CH_3OH (methanol) and $(CH_3)_2CO$ (acetone), and then dried thoroughly in an oven at 105°C.
- After washing and drying, a small brush or suitable wire may be used to dislodge solids from interior surfaces, followed by using compressed, filtered, dry air or N_2 to blow any loose material out of the insert.
- Finally, the glass surface should be deactivated using any recommended procedure.

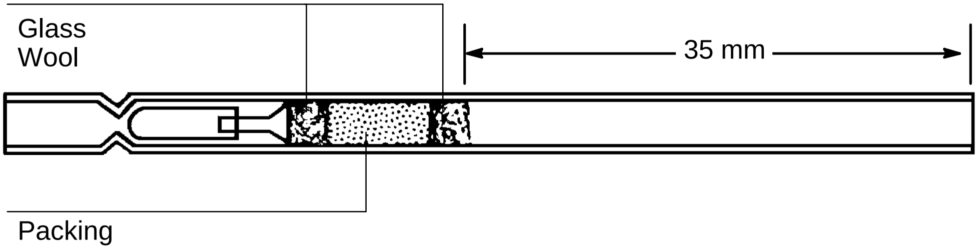
Repacking a split insert

Since, for a split insert, its packing material is discarded in cleaning, the insert must be repacked.

Note: Repacking with small•diameter glass beads is not recommended: they are usually contaminated with metal filings due to sieving procedures used. If they **must** be used, **thorough** cleaning (chemical and physical) is required.

- Use a fresh, small amount of a conventional coated packing such as 2% OV•1 on 100/120 mesh, Chromosorb W•HP.
- Packing is held in place between plugs of silanized glass wool (Part No. 8500•1572).

Figure 8-8



Packing Requirements, Split Insert

- To ensure compatibility with standard 2•inch syringe needles, there should be at least 35 mm between the top of the insert and the top of the packed section.

Metal inserts and/or liners

- Do **not** use concentrated acid(s) on metal inserts or liners!
- The insert is washed with noncorrosive solvents (H_2O, CH_3OH (methanol), $(CH_3)_2CO$ (acetone), CH_2Cl_2 (methylene chloride), etc), and then dried thoroughly in an oven at 105°C.
- After washing and drying, a suitable wire may be used to dislodge solids from interior surfaces, followed by compressed, filtered, dry air or N_2 to blow any loose material out of the insert or liner.
- For any of the inserts used with the dedicated on•columncapillary inlet, use a stainless steel cleaning wire (Part No. 19245•20570; 0.007•inch(0.2 mm) od, 1.5•inch(3.8 cm) long, package of 4) to dislodge solid material.

Flame ionization detector (FID)

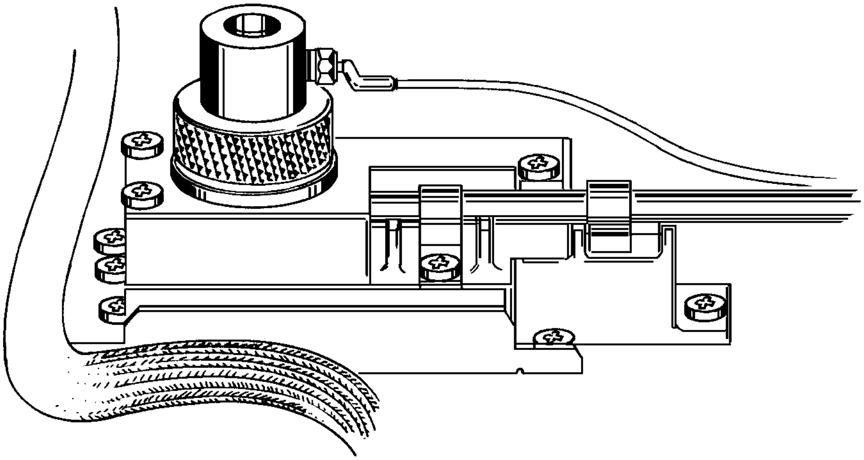
In addition to the detector itself, other systems associated with the detector may also require routine maintenance.

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.

Jet exchange/replacement

Depending upon the column type to be used, and/or analyses to be performed, exchanging the jet in an FID may be necessary.

Figure 8-9



Flame Ionization Detector

Note: The proper jet must be installed prior to column installation.

If switching from packed column operation to capillary operation, the jet for capillary use **must** be installed prior to column installation.

Depending upon the column type to be installed (packed versus capillary), the proper jet must be installed. Table 8•1lists available jets:

Table 8-1. Available FID/NPD Jets

Part No.	Jet Tip ID (inch)*	Use
18789-80070	0.030	Packed Column Only (FID only: Simulated Distillation, TCD-to-FID series operation)
18710-20119	0.018	Packed Column (Standard, FID and NPD)
19244-80560	0.011	Capillary Column (FID and NPD) (FID: high sensitivity, packed column)

---

* Measured at the jet tip.

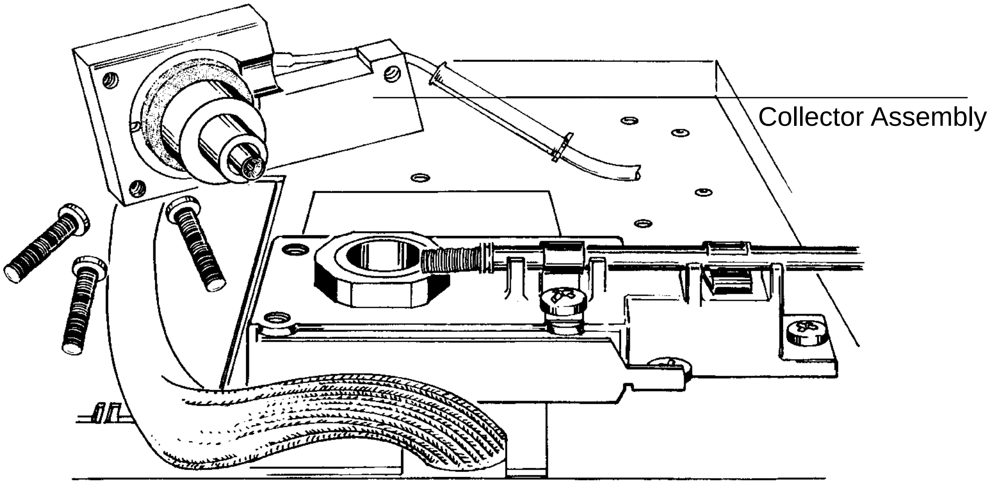
NOTE: The 0.011-inch jet optimizes performance with capillary columns. If used with packed columns, FID flame-out may occur with solvent peaks.

Because jet exchange requires disassembling the collector assembly from the detector base, it is also a convenient opportunity to inspect the detector collector and base for contaminating deposits. Proceed to the next section to disassemble the detector, to exchange the jet, and to perform any necessary cleaning.

Cleaning

Both the jet and the collector bore require occasional cleaning to remove deposits (usually consisting of white silica from column bleed, or black, carbonaceous soot). Such deposits reduce sensitivity and cause chromatographic noise and spikes.

Figure 8-10



Cover Removed, Flame Ionization Detector (FID)

Turn off the detector and its heated zone; also turn off gases to the detector (particularly H_2!). Allow time for the detector zone to cool. Open the top cover at its front edge to access the detector.

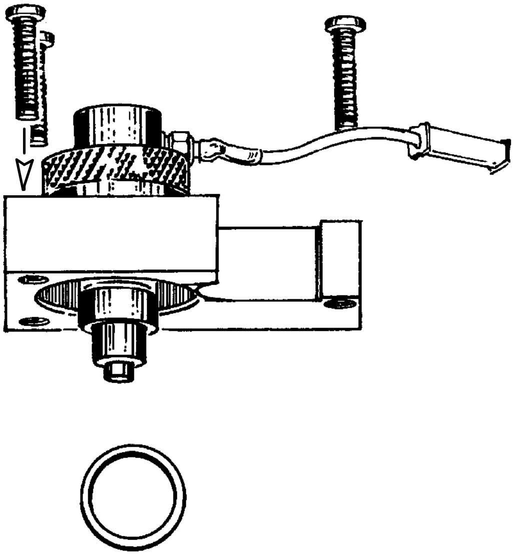
1. Using a Pozidriv•typescrewdriver, remove three screws around the detector cover, and remove the cover.
2. Remove the FID collector assembly by pulling it straight up.
3. Use a cleaning brush (Part No. 8710•1346)to scrub the interior of the collector.

   Using compressed air or N_2, blow out loose material from inside the collector.

WARNING Wear proper eye protection when using compressed gas for cleaning purposes.

Wash the collector in distilled water, hexane, and/or CH_3OH (methanol). Dry in an oven at 70°C for at least 1/2•hour.

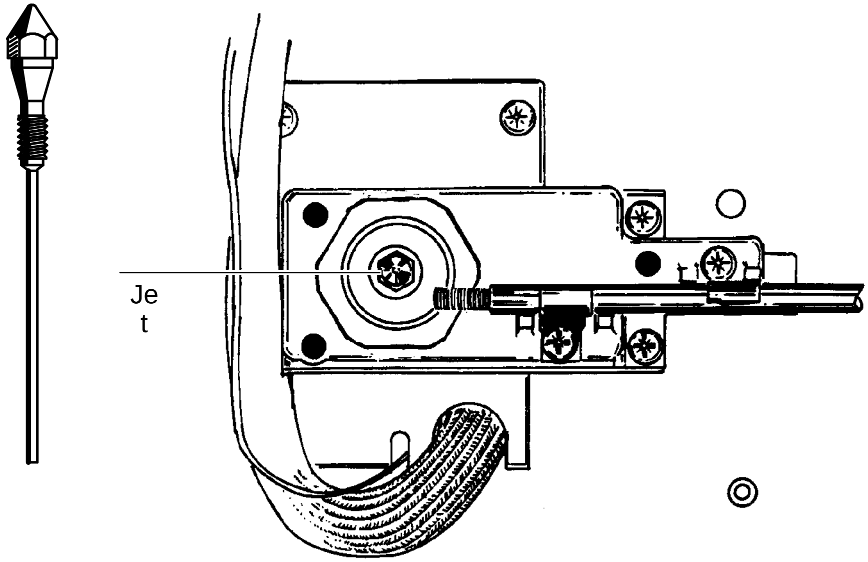
Figure 8-11



FID Collector Assembly

4. Using a 1/4•inchhex nut driver, unscrew (counterclockwise) and remove the jet from the detector base.

Figure 8-12



FID Jet

5. The jet exists in three sizes: 0.030•,0.018•,or 0.011•inch.Use a cleaning wire (0.016•inchod, 12•inchlength, Part No. 18765•20070)to loosen/remove internal deposits. Be careful in using the wire with the 0.011•inchjet.

   Wash both the internal bore and exterior of the jet with a 1:1 (V/V) solution of CH_3OH (methanol) and $(CH_3)_2CO$ (acetone).

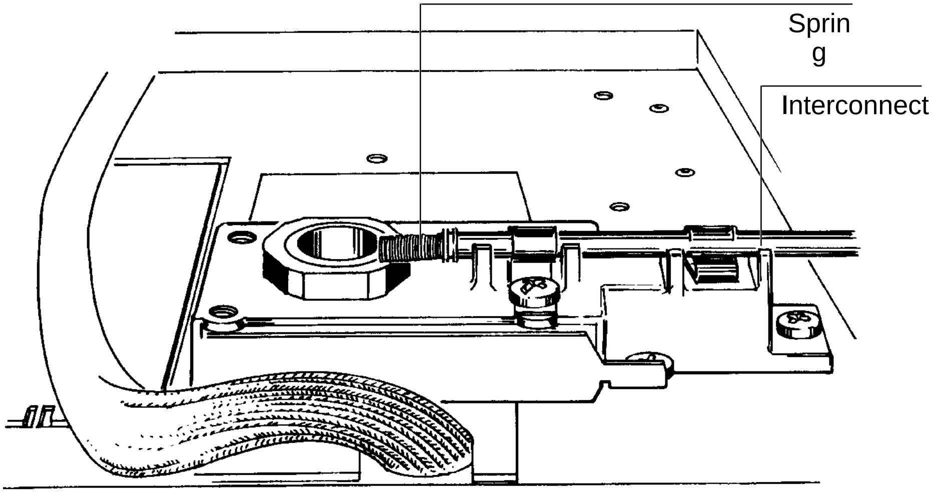
6. Clean the detector base cavity using solvents, a swab, and compressed air or N_2.

7. Reinstall the jet, tightening it to finger•tightness,and then an additional 1/8•turn.

Caution Do not overtighten the jet! Overtightening may permanently deform and damage the jet, the detector base, or both.

8. Replace the collector, being certain the spring contact on the interconnect is in good contact with the groove on the collector.

Figure 8-13



FID Signal Board Interconnect

9. Reassemble the detector cover.

Ignition problems

Before proceeding, make sure that gases are plumbed correctly, the system is leak•free,flow rates are set correctly, and external lines have been well purged.

Note: If Helium is being used as carrier/makeup gas, be aware that flame lighting problems may occur at very high flow rates (> 50 ml/min). If problems arise with He carrier/makeup gas, try using the lowest flow rate possible, consistent with obtaining good chromatography.

Assuming these things are correct, that the detector is contaminant•free, and that the ignitor is operational, if ignition problems persist, a damaged jet may be the cause.

Remove and inspect the jet, looking for galled or scored sealing surfaces, burrs, a bent or crimped tube, etc. Even if no external damage is visible, the jet may be damaged internally by overtightening. For this reason, it is best to have a new jet on hand to exchange if a damaged jet is suspected.

Nitrogen-phosphorus detector (NPD)

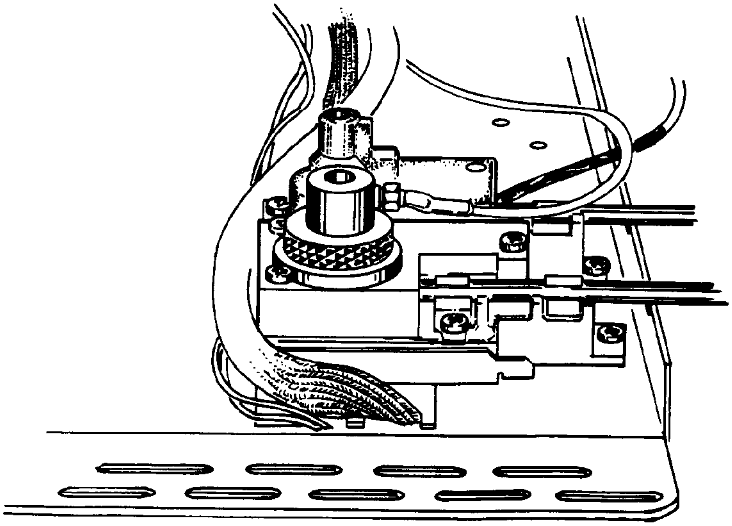
In addition to the detector itself, other systems associated with the detector may also require routine maintenance.

WARNING Nitrogen•phosphorusdetectors 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.

Cleaning

Both the jet and the detector base require occasional cleaning to remove deposits (usually consisting of white silica from column bleed). Such deposits reduce sensitivity and cause chromatographic noise and spikes.

Figure 8-14



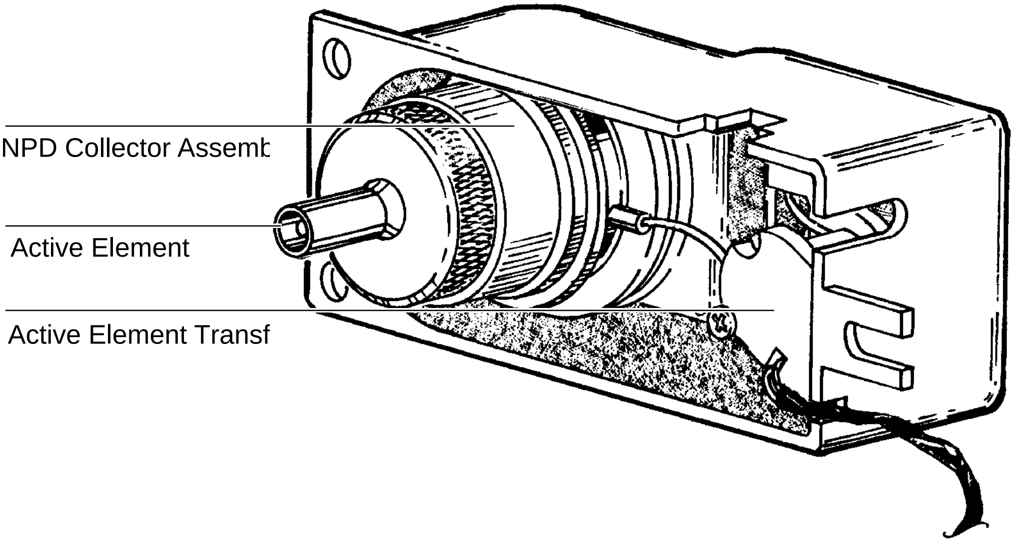
Flame Ionization and Nitrogen-Phosphorus Detectors

Turn off the detector and its heated zone; also turn off gases to the detector (particularly H_2!). Allow time for the detector zone to cool. Open the top cover at its front edge to access the detector.

1. Using a Pozidriv•typescrewdriver, remove three screws around the detector cover and carefully remove the cover (see **Caution** below).

Caution Note that the transformer supplying power to the active element is secured to the inside of the cover. It may be left in place, but be careful not to stress its leads, either those to the collector, or those to the detector signal board.

Figure 8-15



NPD Collector and Active Element Power Supply Transformer

WARNING Wear proper eye protection when using compressed gas for cleaning purposes.

2. a. Using compressed air or N_2, blow out loose material from inside the collector. Do this carefully so as not to disturb the active element.

Caution Do not attempt to clean the inside of the collector by inserting objects such as wires or brushes; to do so may damage the active element.

b. Wash the collector in hexane or isooctane. Then carefully dry the collector using compressed air or N_2.

Caution Avoid polar solvents, especially water; polar solvents may dissolve the rubidium salt coating on the active element.

Note: Avoid touching the lower end of the collector itself (end nearest the jet); fingerprints and/or other contamination may cause baseline drift and noise.

3. If worn or cracked, replace the collector sealing gasket (Part No. 0905•0915;see Figure 8•11).
4. Using a 1/4•inchhex nut driver, unscrew (counterclockwise) and remove the jet from the detector base (see Figure 8•12).
5. The jet exists in two sizes: 0.018•or 0.011•inch.Use a cleaning wire (0.016•inchod, 12•inchlength, Part No. 18765•20070)to loosen/remove internal deposits. Be careful using the wire with the 0.011•inchjet.

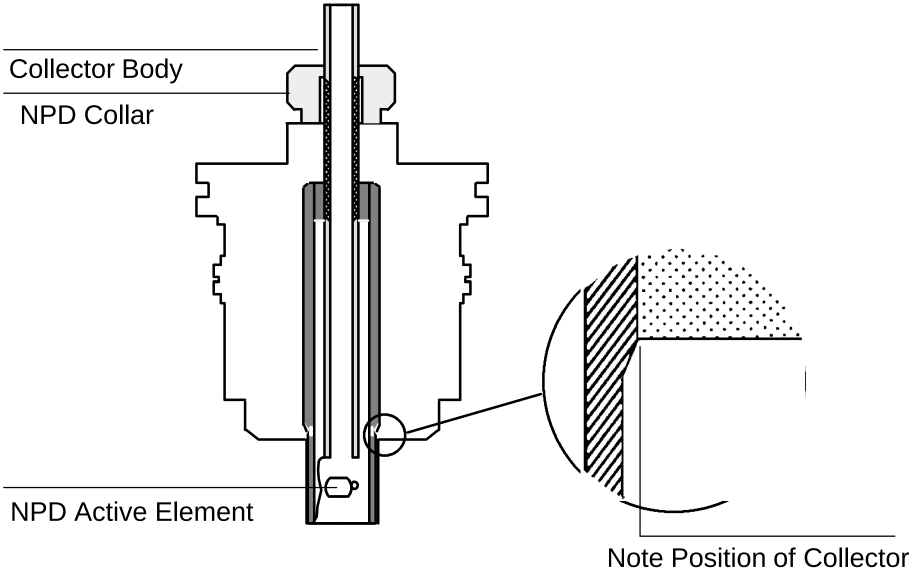
   Wash both the internal bore and exterior of the jet with a 1:1 (V/V) solution of CH_3OH (methanol) and $(CH_3)_2CO$ (acetone). Dry the jet with compressed, clean air or N_2 and heat in an oven at 70°C for at least 1/2•hour
6. Clean the detector base cavity using solvents, a swab, and compressed air or N_2.
7. Reinstall the jet, tightening it to finger•tightness,and then an additional 1/8•turn.

Caution Do not overtighten the jet! Overtightening may permanently deform and damage the jet, the detector base, or both.

8. Replace the NPD collector, and transformer and cover assembly. Be certain the spring contact to the signal board is in good contact with the groove on the collector (see Figure 8•13).During reassembly do not touch the lower portion of the collector assembly because fingerprints and/or other contamination may (will) cause baseline drift and/or noise.

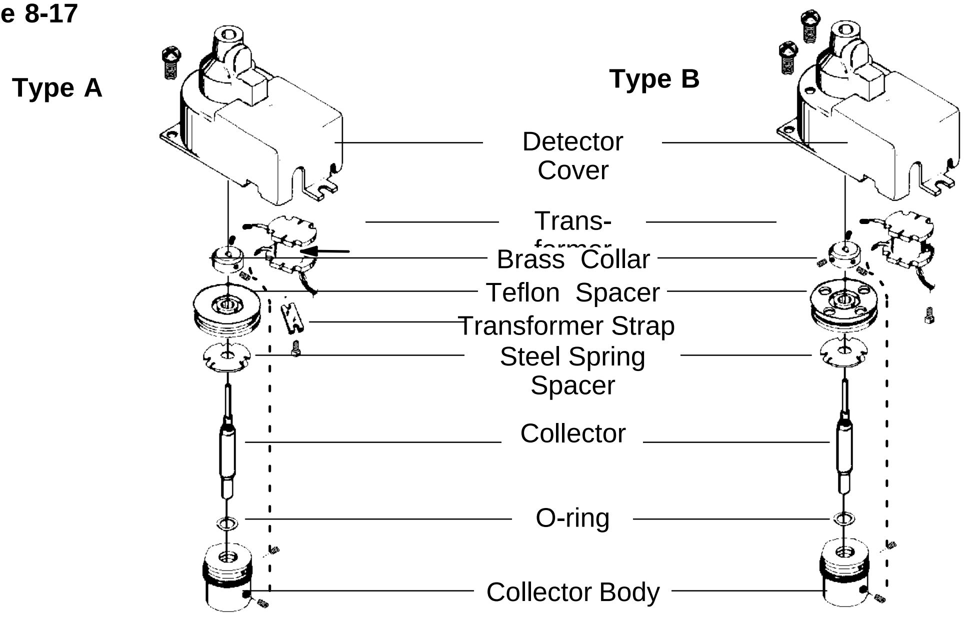
Removing/replacing the NPD collector

Figure 8-16



Cross-Section, Assembled Type A NPD Collector

Figure 8-17



NPD Collector and Collector Assembly

Whenever the collector must be removed from the detector cover of a Type A NPD, the following procedure should be used:

Note: During disassembly do not touch the lower portion of the collector assembly. Use clean, lint•free gloves to prevent contamination of the assembly. Suitable gloves (HP Part No. 8650•0030) are available.

1. Following the procedure under *Cleaning*, remove the collector assembly from the detector base. A 1.5•mm hex wrench is required for disassembly.
2. Loosen the setscrew in the brass collar at the top of the collector assembly. Remove the transformer lead from the collar; then remove the collar itself.

   The remaining transformer lead does not need to be disconnected to remove the collector. However, if desired, it is easily removed by loosening the setscrew in the stainless steel portion of the collector body.

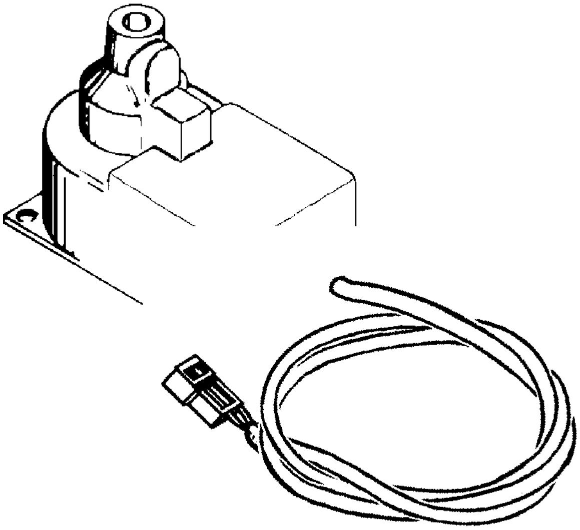
3. Remove the Teflon spacer and stainless steel spring spacer from the top of the collector body.
4. Loosen the setscrew in the Teflon portion of the collector body.
5. Grasping the collector at its top end (to avoid contaminating its detecting end), withdraw it from the collector body. Some resistance to this occurs until the O•ringseal becomes free.
6. Noting the following, reassemble in the reverse order:
    - The O•ringseal should be placed onto the collector. The collector is then inserted into the collector body from the top. Insert it only far enough to extend a few millimeters past the bottom face of the collector body.
    - The stainless steel spring spacer must be installed with its tabs **toward** the collector body (against the stainless steel portion).
    - The Teflon spacer must be mounted with its flat (ungrooved) side **toward** the collector body (against the stainless steel spring spacer).
7. Inspect the brass collar; note that the hole through it is slightly larger on one side than the other. Install the brass collar onto the end of the collector so the side with the **larger** hole is **toward** the collector body.
8. Press the assembly together firmly by pressing down on the brass collar while holding the collector body. Make sure the sealing O•ring has seated itself into the collector body.
9. While holding the assembly together, note Figure 8•16to verify the collector is correctly positioned. If so, tighten the setscrew in the collector body to secure the collector.

    If the collector is extended too far beyond the lower end of the collector body, it is likely the brass collar is installed upside down. Reposition the collector in the collector body, reinstall the brass collar, and press the assembly together again.
10. While holding the assembly together, replace the transformer lead into the brass collar (loosening the setscrew further may be necessary). Orient the collar so the lead is adjacent to the transformer

lead on the collector body. Tighten the setscrew to secure the wire and collar.

Type B NPD transformer/collector assembly

Figure 8-18.



Type B NPD Detector Assembly

Remove the transformer/collector assembly from the Type B NPD cover as follows:

Caution During disassembly do not touch the lower portion of the collector assembly. Use clean, lint•freegloves to prevent contamination of the assembly. Suitable gloves (HP Part No. 8650•0030)are available.

1. Remove the two screws holding the transformer inside the cover.
2. Remove the two screws holding the collector insulator to the cover. These are removed from the top side.
3. Slide the transformer wires through the slot in the cover by maneuvering the uninsulated portion of the wires to the slotted hole.

4. Remove the collector from the collector assembly as follows:
    - Loosen the 1.5•mmscrew holding the transformer secondary wire to the top of the collector and disconnect the wire. The hex key wrench required is a 1.5•mmsize and was provided with the instrument.
    - Loosen the 1.5 hex key screw holding the brass connector to the collector top and remove the brass connector.
    - Remove the Teflon spacer and the stainless steel spacer/spring from the top of the collector body.
    - Loosen the 1.5•mmhex key screw holding the collector into the collector body and pull the collector out the top of the collector body. Grasp only the top portion of the collector to avoid contamination. If you plan to reinstall the collector, lay it aside on a clean surface until ready for reassembly. See below for cleaning instructions.
5. The jet can be removed for cleaning or replacement (if the jet is very dirty it should be replaced) with the 1/4•in.nutdriver supplied with the instrument. Using the nutdriver, unscrew (turn jet counterclockwise) and remove the jet from the detector base.
    - The jet exists in two sizes: 0.018•in.and 0.011•in.id. A cleaning wire may be used to help remove deposits from the bore of the jet (jet cleaning wire, Part No. 18765•20070is 12 inches of 0.016 od wire that can be used for this purpose).
    - Wash both the bore and the exterior of the jet with a 1:1 (V/V) solution of methanol and acetone. The jet may also be cleaned in an ultrasonic bath in the same solution. It is best to dry the jet and bake it out in an oven (70 to 150°C) before replacing it in the detector. After cleaning, apply a small amount of lubricant to the threads of the jet. The lubricant is necessary to prevent galling of the threads in the detector base.
    - The detector base may be cleaned using solvents (such as used for the jet), a cleaning swab, or wire brush, and dried with low•pressure compressed air.

Caution

The detector should be cold and the instrument unplugged to avoid the danger of the flammable solvents flashing and possibly igniting the fumes.

Reinstallation

1. Reinstall the jet in the detector base (using a 1/4•inchnut driver). Make sure that the threads are clean and free of burrs that could cause damage. If there is any binding, the cause should be determined and corrected before proceeding. If a torque wrench is available, no more than 1 Nm (newton meter) of torque should be used to install a new jet.

Caution Do not overtighten the jet! Overtightening may permanently damage the jet, the detector base, or both.

2. Cleaning and reassembly of the collector/collector assembly is as follows:

   - Powdery deposits may be removed from the interior of the collector by gently blowing out the loose material with low•pressurecompressed air or nitrogen.

Caution Do not attempt to clean out the inside of the collector by inserting objects such as wires or brushes•to do so may damage the active element.

   - Inspect the collector assembly to make sure that the active element is centered in the tube and is not shorted to the side and that it is positioned horizontally.

- All collectors should be washed off with GE grade hexane or a similar solvent before reinstalling in the instrument to remove any grease, fingerprints, or other contaminants. Soak the entire collector in a vial of hexane for several minutes (2-10). Remove the collector, touching only the top portion, and scrub the lower, outer collector tube with a clean wipe soaked with hexane. Dry off the excess hexane solvent.
- Place a new O•ringon the collector and place it into the collector body assembly from the top. Carefully position the collector tube so that the bevel on the outer tube is positioned properly in relation to the bottom edge of the collector body. Tighten the collector holding screw firmly against the collector tube. This connection is both a mechanical connection and an electrical contact.
- Complete the collector assembly as follows: Wear clean, lint•freegloves to avoid contaminating the collector assembly; (1) make sure the O•ring is depressed into the groove in the collector body; (2) examine the collector sealing gasket for wear or cracks and replace if necessary; (3) place the stainless steel spacer/spring onto the collector/body assembly; (4) place the Teflon spacer on top of the spring•the side with the groove faces up; (5) if necessary, replace the bottom transformer secondary wire into the hole in the collector body and tighten the setscrew onto the wire connector.
- Attach the brass collar to the top of the collector tube and tighten the collar•holdingscrew. The smaller hole in the brass collar faces up.
- Insert the top transformer secondary wire into the slot in the brass collar and tighten the wire•holdingscrew on the bare wire. This must be a good electrical connection, but take care and do not completely cut the wire with the screw.
- Carefully position the transformer/collector body assembly into the cover and fasten the Teflon insulator of the collector body in place with the two screws. Do not overtighten the screws into the Teflon and strip the threads.
- Carefully position the transformer with both end covers in place between the threaded standoffs and fasten in place with the two screws. Place the assembled detector cover in place onto the detector base and fasten in place with the three screws.

Electron capture detector (ECD)

Frequency test

Note: For high sensitivity operation, and starting from a cold system, 24 hours may be necessary before baseline is completely stabilized. Use low•bleedsepta and condition a new septum prior to use in an unused inlet for several hours with 1 to 5 ml/min carrier flow rate.

Using normal operating conditions, and allowing at least two hours after the last sample was injected, enter the following:

`DET` `A` (or `B`) `ON`

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

If the displayed value exceeds 100 (1000 Hz), indicating a high background signal, investigate the following possibilities:

- Contaminated carrier gas trap(s) and/or supply
- Insufficient column conditioning
- Contaminated detector
- Column, inlet, and/or septum bleed
- Leaks
- Anode insulation leakage

Carrier gas evaluation

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

The following procedure should be performed whenever a new carrier gas source is installed:

1. With the instrument on and operating normally, cool the oven to ambient, turn off the detector, turn off carrier flow to the detector, and

remove the column to the ECD. If a capillary column was installed, remove also the makeup gas adapter in the detector base.

2. Disconnect the carrier gas source line at its fitting on the HP 5890.
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 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. Enter the following:

   DET A (or B) ON

   SIG 1 (or SIG 2) (A or B)
7. Within 15 minutes, displayed signal values should be within the range 40 to 100 (400 to 1000 Hz); there may be downward drift.
8. If displayed values are greater than this range, trap(s) may be at fault. Connect the carrier gas supply line directly to the detector base and repeat the test.

If values are now within range, trap(s) are likely at fault; if still out of range, the carrier gas supply itself may be contaminated.

Leaks

Note: This test assumes that flow system components upstream from the detector (inlet, column) are leak•free.

1. Set inlet, oven, and detector temperatures to ambient and allow time for cooling. Turn off the detector, and turn off its carrier flow.
2. Use a vent plug (Part No. 5060•9055)to cap the ECD exhaust vent.
3. Set carrier gas pressure at the inlet to the instrument to 103 kPa (15 psi). 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 carrier gas flow at its source and monitor system pressure.
5. The system may be assumed to be leak•free if no pressure drop is observed over a 10•minute period.
6. If leakage is observed, use an appropriate leak•detecting fluid to check for leaks at detector column fittings and at the plugged vent.
7. The detector body itself is **not** a likely source of leaks; in any case, it cannot be disassembled without special license from the Nuclear Regulatory Commission or Agreement State Licensing Agency (USA only).

Thermal cleaning

Caution

Detector disassembly and/or cleaning procedures other than thermal should be performed only by personnel trained and licensed appropriately to handle radioactive materials. Trace amounts of radioactive ^{63}Ni may be removed during other procedures, causing possible hazardous exposure to β•and X•radiation (bremsstrahlung).

Cleaning by **bake•out** of the detector is performed only after verification that carrier supply gas and flow system components, are leak•and contaminant•free.

WARNING

To prevent possible hazardous contamination of the area with radioactive material, the detector exhaust vent always must be connected to a fume hood, or otherwise vented in compliance with the latest revision of Title 10, CFR, Part 20, or with state regulations with which the Nuclear Regulatory Commission has entered into an agreement (USA only). For other countries, consult with the appropriate agency for equivalent requirements.

Packed column:

1. Close the anode purge on/off valve.
2. Remove the column from the detector; install in its place an empty glass column.
3. Establish normal carrier gas flow rate (20 to 30 ml/min); set oven temperature to 250°C.
4. **Open** the anode purge on/off valve.
5. Heat the ECD to 350°C. Allow thermal cleaning to continue for 3 to 12 hours. If the displayed value does not drop below 60 (600Hz), indicating a high background signal, it is recommended that the detector be returned to Hewlett-Packard for cleaning.

Capillary column:

1. Close the makeup gas and anode purge on/off valve.
2. Remove the column from the makeup gas detector.
3. Install a paper clip or similar gauge wire in the same manner as a capillary column.
4. Open the makeup gas on/off valve and establish a makeup gas flow rate of 50 to 60 ml/min.
5. Open the anode purge on/off valve.
6. Set the oven temperature to 250°C.
7. Heat the ECD to 350°C. Allow thermal cleaning to continue for 3 to 12 hours. If the displayed value does not drop below 60 (600Hz), indicating a high background signal, it is recommended that the detector be returned to Hewlett•Packardfor cleaning.

Radioactivity leak test (wipe test)

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

The procedure used is the **wipe test**. A wipe test kit (Part No. 18713•60050)is supplied with each new ECD. Its contents are listed in Table 8•2:

Table 8-2. HP 5890 Radioactivity Leak Test (Wipe Test) Kit

Item	Description	Part No.	Qty
1	Envelope	05750-80036	4
2	Envelope	05890-90920	4
3	Information Card	18713-90040	12
4	Filter Paper, Circular	3150-0429	12
5	Service Note	5950-3586	1
6	Plastic Bag	9222-0308	12

Thermal conductivity detector (TCD)

Cleaning

Cleaning by **bake•out** of the detector is performed only after verification that carrier supply gas and flow system components, are leak•and contaminant•free.

1. **Turn off the detector!**

   DET A (or B) OFF

2. Remove the column from the detector; cap the detector column fitting.

Caution Failure to turn off the TCD and to cap the detector column fitting may cause irreparable damage to the filament due to O_2 entering the detector.

3. Establish normal reference gas flow rate (20 to 30 ml/min) through the detector (set oven temperature to 250°C).
4. Heat the detector to 400°C; allow thermal cleaning to continue for several hours.

Flame photometric detector

Cleaning/replacing FPD windows, filters, seals

Column bleed and/or effluent can contaminate the first quartz window (heat shield) nearest the detector module. Dust, fingerprints, atmospheric contaminants can dirty both quartz windows, the filter, and/or the photomultiplier tube (PMT) window. Contamination anywhere along the light path between flame and PMT can reduce detector sensitivity.

Turn the detector off.

Turn off hydrogen, air (or oxygen), and auxiliary nitrogen supplies to the detector (manifold on•offvalves); for convenience, carrier flow and other temperature zones may be left on and at their respective setpoints. Allow time for the detector module to cool.

Remove the PMT assembly from the detector module and remove the filter. Use lint•freelens tissue to clean the filter (both sides) carefully, and the PMT window seen inside the housing. Be careful not to scratch surfaces; do not use any cleaning fluid that might leave a film upon drying.

Inspect the filter; chips, scratches, and/or cracks in the light path scatter light, reducing detector sensitivity. Replace filter(s) as necessary (sulfur mode filter, Part No. 19256•80000;phosphorus mode filter, Part No. 19256•80010).

Likewise, damage to the PMT window cannot be tolerated; if necessary, replace the PMT or call Hewlett•Packard support.

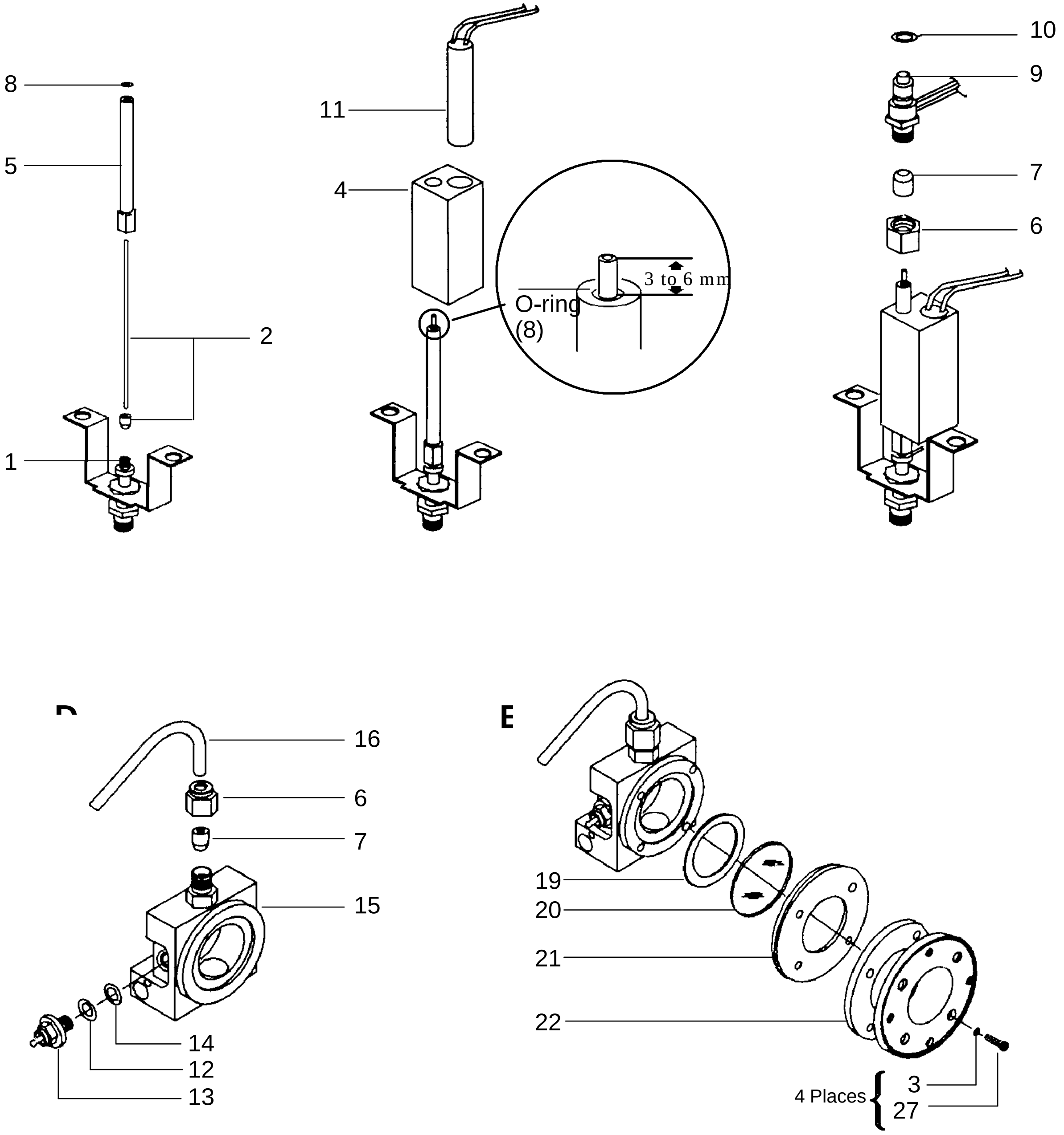
1. Remove four screws to remove the PMT adapter flange. Remove the adapter carefully; a quartz window is exposed and may fall out. The window is cleaned in a manner similar to the filter.
2. Remove four more screws to remove the stainless steel coupling. Remove the coupling carefully; the remaining quartz window may fall out. Clean the window in a manner similar to the filter.

Note placement and types of seals found on disassembled parts. Seals should be replaced on reassembly. (See Figure 8•21 for seal part numbers.)

Inspect the windows; chips missing, scratches, and/or cracks in the light path scatter light, reducing detector sensitivity. Replace window(s) if necessary (first window•Part No. 19256•80030; second window•Part No. 19256•80060.)

Reassemble parts in reverse order, making sure all seals are of the proper type and in their respective proper locations. Tighten screws firmly to ensure gas•tight and light•tight seals. If the filter has a silvered side, it should face the flame (indicator arrows (>) on edge of filter should point toward the PMT).

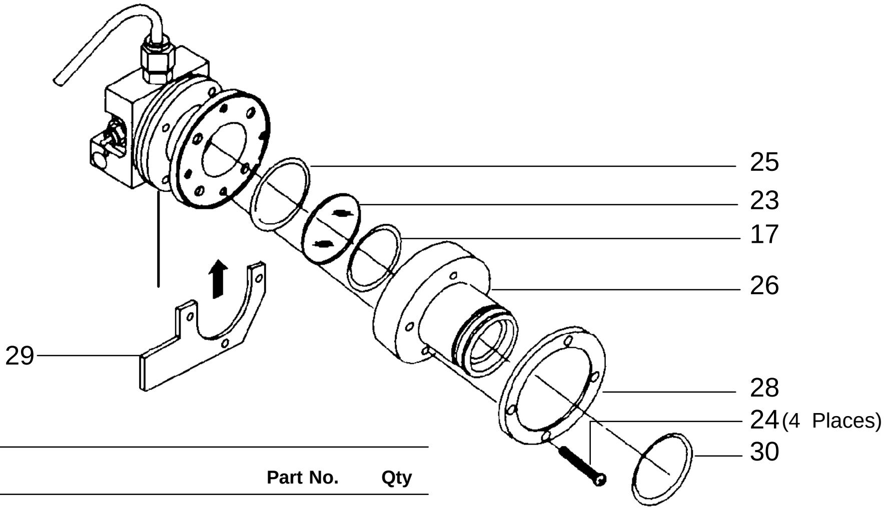
Figure 8-20.



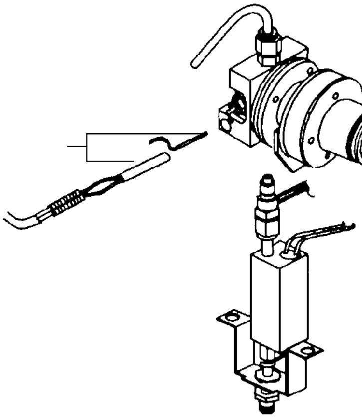
Subassembly Parts Identification

Figure 8-21.

F



Item	Description	Part No.	Qty
1	Weldment, Base	19256-80540	1
2	Gigabore Liner/Ferrule Assembly (see note)	19256-60590	1
3	Lockwasher	2190-0108	4
4	Lower Heater Block	19256-20500	1
5	Weldment, Transfer Tube	19256-80550	1
6	Nut, Brass, 1/4-inch id	0100-0056	2
7	Ferrule, Vespel, 1/4-inch id	0100-1061	2
8	O-ring-Kalrez, Transfer Tube	0905-1101	1
9	Weldment, Jet	19256-80580	1
10	O-ring-Kalrez, Jet Cartridge	0905-1103	1
11	Heater/Sensor Assembly	19256-60540	1
12	Spacer, Ignitor	19256-20590	1
13	Glow Plug	0854-0141	1
14	O-ring-Kalrez, Ignitor	0905-1102	1
15	Weldment, Block	19256-80560	1
16	Exit Tube Assembly	19256-20700	1
17	O-ring, Orange, 1.05-inch id (Silicone)	0905-1104	1
18	Drip Tube, Silicone Rubber	19256-20730	1
19	Gasket, Head Shield	19256-80040	1
20	Window, First Heat Shield	19256-80030	1
21	Disk, Heat Shield	19256-20580	1
22	Coupling, Stainless Steel	19256-20550	1
23	Window, Second Heat Shield	19256-80060	1
24	Screw, M3 x 25 mm long	0515-0065	4
25	O-ring, Orange, 0.926-inch id (Silicone)	0905-0955	1
26	Flange Adapter	19256-20510	1
27	Screw, M3 x 12 mm long	0515-0911	4
28	Flange Ring	19256-00200	1
29	Clamp	19256-00090	1
30	O-ring, Brown, 1.239-inch id (Viton)	0905-1100	1



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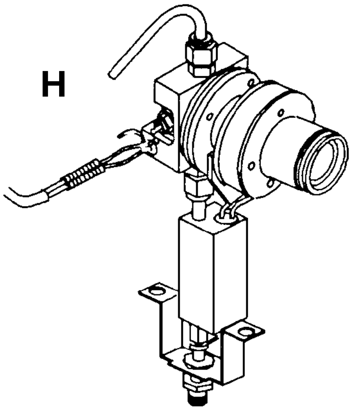


Subassembly Parts Identification

NOTE: Once installed, the ferrule cannot be removed from the liner for reuse unless both parts are still warm.

Cleaning/replacing the FPD jet

If a response problem is encountered (sensitivity, noise, selectivity), the FPD jet should be inspected for deposits and, if necessary, cleaned or replaced. To service the jet properly, the detector module should be removed from the instrument, followed by appropriate service:

1. Turn off power to the gas chromatograph and disconnect the main power cord. Remove the detector covers.
2. Turn off hydrogen, air (or oxygen) and auxiliary nitrogen supplies to the detector (manifold on/off valves). For convenience, carrier flow may be left on. Allow time for heated zones to cool to safe temperatures.
3. Remove the photomultiplier tube (PMT) assembly from the detector module; also remove the filter. Set both in a safe place. Also, remove exhaust tubing (1/4•inchswage fitting) and the chimney assembly (two M4 screws on sides). Then loosen the **U** clamp that locks the burner to support bracket (Part No. 19256•00080), and disconnect the jet assembly from the transfer line tube. Carefully lift the detector module vertically from the transfer tube so as not to damage the fused silica liner. See Caution and WARNING notes which follow.

   Note: It is unnecessary to disconnect any plumbing, ignitor leads, or the heater/sensor. Leave all attached and disconnect the detector block from the transfer line at the 1/4•inchswage fitting; then gently lift block and rotate it enough to access the jet.
4. Remove and inspect the jet assembly. Use a suitable wire to remove any deposits.
5. This is also an ideal time to inspect/clean the glow plug (see *Flame ignition problems*) and inspect/clean the quartz windows (see **C***leaning/replacing windows, filter, seals*).

6. Use compressed gas, air, or N_2 to blow out loose particles from the jet and/or detector module body.
7. Inspect and clean deposits from the jet bore and from the threads using a suitable wire. If the jet is damaged in any way, it should be replaced. It is good practice to replace the jet rather than cleaning it, particularly when extremely high sensitivity is required.
8. A new Kalrez O•ringseal (Part No. 0905•1103) must be used when reinstalling the jet into the burner chamber.
9. Reassemble all parts of the detector module; reassemble the module onto the instrument. A new Vespel ferrule (6.4 mm id, Part No. 0100•1061)should be used to seal the detector module to the transfer line.

Caution Be careful not to crush or side load the fused silica liner when reinstalling the detector.

WARNING Whenever working with exposed fused silica tubing, wear eye protection; fragments of fused silica could be released if the fused silica is fractured or crushed.

10. Reinstall the PMT assembly on the detector module; restore instrument gases and power.

FPD leak testing (GC with electronic flow sensor)

If the system has an electronic flow sensor (EFS) with any FPD gas plumbed through it (air, oxygen, hydrogen, or carrier), the system can be checked easily and quickly. First, close all supply gases except for the one plumbed through the EFS. Then cap off the detector exhaust tube with a 1/4•inchSwagelok plug (Part No. 0100•0196)and a 40% graphitized Vespel ferrule (Part No. 0100•1061).

With the flow system deadheaded and one pressurized gas plumbed through the EFS, the flow reading should drop very close to zero. If not, this indicates a leak in the system. Begin checking possible leak sources and monitor the EFS to determine when the leak has been eliminated.

Possible leak sources, in order of probability are:

1. septum
2. column fittings
3. supply line swage•typeplumbing connections
4. detector block O•ringor Vespel seals
5. other system plumbing

Caution When leak•testingthe flow system under pressure, do not exceed 210 kPa (30 psig), because higher pressures may damage the detector block window or seals.

FPD leak testing (GC without electronic flow sensor)

If a leak is suspected in an instrument without an electronic flow sensor, possible leak sources can be checked in the order listed (1 through 5) above. This can be done by capping off the detector exhaust and pressurizing the system to 140 kPa (20 psig). Then close off the supply flow and monitor the rate of pressure drop. Pressure should drop no more than 7 kPa (1 psig) per minute.

WARNING If using liquid solution to locate leaks, to avoid a potential shock hazard, place the main power switch in its off position and disconnect the main power cord. Be careful not to spill leak solution on electrical leads•especially the detector heater leads!

Note: Except when leak testing, it is best to leave the detector module at operating temperature at all times (whether the flame is lit or extinguished) to minimize thermal expansion/contraction.

Conditioning chemical traps

Remove the trap from its installed location and attach it to a clean, dry gas source (helium or nitrogen). Attach the 1/8•inch end (male) of the chemical trap assembly to the reconditioning gas source using a graphite or a graphitized Vespel ferrule (Part No. 0100-1107) and swage nut (Part No. 0100-0058), as metal type ferrules will damage the sealing surface of the 1/8•inch adapter. Remove the O•ring (Part No. 5180•4181), as the conditioning temperature will destroy it. Set oven temperature to 300°C. Establish a purge gas flow of 60 to 100 ml/min overnight (sixteen hours).

9

Chromatographic Troubleshooting

Chromatographic Troubleshooting

Introduction

This chapter is concerned with diagnosis: the process of going from unexpected behavior of the HP 5890 SERIES II (hereafter referred to as HP 5890) (symptoms) to the probable location of the difficulty (causes).

Problems arise from many causes. Some of these are:

- Electronic or mechanical failure
- Contamination in critical areas, such as detectors
- Incorrect or inappropriate setpoints
- Leaks, column or septum bleed, or other chromatographic difficulties

These may interact to a bewildering degree: For example, baseline problems may arise from any of the above areas. Accordingly, this chapter is organized by symptoms, with reference to most probable causes.

Baseline symptoms

Position

1. Baseline not at left (lower) part of chart:
   - Check the zero of your recording device: An attenuation or range change during the run may be responsible.
   - Check TCD signal polarity.
2. Baseline position changes suddenly during the run:
   - This usually results from a range or attenuation change.

- It can also result from valve operations: If valves are being switched during a run, examine the valve time program to see if the change coincides with a valve operation.
- This symptom also can occur if the septum suddenly begins to leak; Avoid the problem by changing septa regularly.
- Offset•see troubleshooting procedure for the particular detector in use.

Wander and drift

Baseline wander or drift may occur when a flow or temperature setting is changed. If the system is not stabilized at the new conditions before starting a run, some baseline changes are to be expected. Cases following assume that sufficient stabilization time has elapsed since the last change in operating conditions.

Wander and drift are often accompanied by noise, discussed below.

1. Baseline moves steadily upscale or downscale (drift) during the run:
    - This is most frequently seen during temperature programming: Operation with a single column (no column compensation) at moderate to low attenuation causes this. If dual columns are used, check that the signal mode is correct for column compensation.
    - It is also possible the compensation is insufficient (upscale movement) or too great (downscale movement).

      This cause of drift is minimized by thorough column conditioning. Operating at lower oven temperature also helps but prolongs the analysis. Use of a chromatographically equivalent column with higher temperature limit is also possible.

2. Baseline is erratic, moves up and down (wander):

   - Suspect a leak in the system: Check septum condition and replace if necessary. Check column connections.

     If the leak is at the detector end of the column, retention times are stable from run to run, but sensitivity is reduced. If it is at the inlet end, sensitivity is reduced and retention times are delayed.

Noise

Noise is rapid vertical baseline fluctuations, broadening the baseline and giving it a hairy appearance. Noise is different from spiking; spikes are isolated events, rather than almost continuous, and are described on the next page.

Some noise is inevitable with any detector. At high attenuation it is invisible but appears as attenuation is decreased. Noise limits useful detector sensitivity; thus, it should be minimized.

1. Noise appears suddenly on a previously clean baseline:

   - Consider all changes made recently in the system: Reduced attenuation, for example, makes noise more apparent, though the absolute noise level is unchanged.

   - New septa may contribute noise from bleed of low molecular weight material. If noise decreases when inlet temperature is lowered, this is a likely cause. Only highest quality septa should be used.

   - Contaminated carrier gas: If a tank was replaced recently, and the old one is available and has some gas left in it, try the older tank to determine if noise decreases.

     If the new gas is so badly contaminated it saturates traps, changing to the old gas may not show improvement until traps are regenerated. This problem is most common with N_2 carrier gas, since suppliers may exercise less care than with other common carrier gases.

- Contaminated detector gases (hydrogen and air).
- Air currents from a fan or air conditioner blowing across the top of the instrument may interfere with gas exiting from the detector.

  This is a possible, though not very likely, cause of noise since detectors are well protected. Switching off the air current source or shielding the detector area identifies this problem.
- An inadequately tightened collector on an FID or NPD generates noise.
- A contaminated detector results in noise.

2. Noise increases gradually to an unacceptable level:

   - This symptom indicates gradual buildup of the noise source, rather than an abrupt change as discussed above. FI detectors are susceptible to gradual buildup of deposits in the collector. In extreme cases spiking occurs along with increased noise level.

     Silicon dioxide deposits are formed when bleed from a silicone column is burned in the flame. This material is removed mechanically. Preventive measures include use of low column loadings, stationary phases with high•temperaturelimit, thorough column conditioning before use, and the lowest possible oven temperatures for the analysis.
   - Carbon deposits may form from solvents that burn poorly (primarily chlorinated materials and aromatics). If possible, avoid such solvents. If they are necessary, periodic cleaning of the collector is required.
   - Gradual noise increase may occur from saturated carrier gas drier or chemical traps. When these approach their capacities, contaminants begin to pass through and create noise. Trap and drier regeneration or replacement eliminates this source of noise.

Spiking

Spikes are isolated baseline disturbances, usually as sudden (and large) upscale movements. If accompanied by noise, the noise problem should be solved first, since spiking may disappear at the same time.

1. Spikes appear whenever the chart is running:
   - The cause is almost always electronic in origin: Loose connections are likely. Check signal cable connections at the detector and controller ends.
   - A dirty slidewire on a recorder may cause this; see the maintenance section of your recorder manual.
   - Loose or dirty contacts between printed circuit boards and their connectors may be responsible. Read appropriate sections regarding servicing boards and connectors for the HP 5890.
2. Spikes appear on chromatograms but not when the recorder is isolated (no input signal):
   - This indicates a detector problem: In extremely dirty FID collectors, particles may break away, causing disturbances in the most sensitive area of the detector. This appears on the chart as a spike.
   - A less common cause is column packing particles being blown into the detector: A loose or inadequate glass wool plug at the detector end of the column may fail. As particles are blown through the detector, the disturbance generates a spike. This may happen with any detector, but FIDs and NPDs are particularly susceptible because of the narrow bore of the jet.

     The bottom of the jet in FIDs and NPDs extends into the end of a packed column. If the bottom touches the glass wool plug at the end of the packing, spikes are produced.

Retention time symptoms

Retention time drift

Retention time drift is a steady increase or decrease of retention times in successive runs. Erratic times (both directions) are discussed below as retention time wander.

1. In a series of runs, retention times suddenly increase:
   - This may be due to an oven temperature change or to change in flow; verify setpoints.
   - A **blown** septum is a possibility. If this happens, the change is probably at the beginning of a run.
   - The carrier gas tank may be nearly empty.
2. In a series of runs, retention times suddenly decrease:
   - This is likely to be due to a setpoint change, either in oven temperature or in carrier gas flow rate; verify both.

Retention time wander (reproducibility)

1. Retention time reproducibility is erratic throughout the run:
   - If the runs are made with manual injection, the suspect is injection technique. Variation in time between sample injection and pressing START causes variation in retention times. With automatic injection this possibility is minimized.
   - Oven temperature variation may cause this; monitor oven temperature during a run to check this.

2. Reproducibility is good early in the run but not toward the end:
    - This may occur in temperature•programminga very densely packed column; as column contents expand with heating, resistance to flow may be so great that a mass flow controller cannot maintain constant flow. Try increasing carrier source pressure.

      If this is the cause, the problem will either vanish or its onset will move later in the run.
    - This may indicate too low a starting temperature; many stationary phases have a minimum operating temperature, usually the melting point of the material. Below this temperature, gas•solid chromatography is performed; above it, gas•liquidchromatography is performed. If an oven temperature program passes through this range, results can be very erratic.
3. Reproducibility is good later in the run but not for the first few peaks:
    - When earliest peaks elute very rapidly, they may not have had time to achieve chromatographic equilibrium with the stationary phase; they act like solvent peaks and are blown straight through the column.

      A useful rule is that peaks of interest should require at least four times as long to appear as an unretained solvent or air peak. If this problem is suspected, try reducing oven temperature 30°C to approximately double the retention times.
4. Retention time changes with amount of sample:
    - Sample is overloading the column: When there is more sample than the stationary phase can handle, peaks will be deformed and shifted from correct retention times.

      In gas•liquidchromatography an overloaded peak shifts to a longer retention time and tails on its trailing edge. With gas•solid chromatography the effect is the opposite. Try diluting the sample or injecting less.

Peak symptoms

No peaks

This is usually due to operator error; possibilities include injection on the wrong column, incorrect signal assignment, attenuation too high (peaks are present but not visible), a bent syringe needle in an automatic sampler, etc. Check system parameters for the analysis.

Inverted peaks

This is likely an inappropriate signal assignment definition (e.g., B - A with sample injected on column A) or incorrect polarity with a TCD.

Extra peaks

These are divided into two classes: **Additiona**l peaks appear on the chart in addition to those expected from the sample. **Ghost** peaks appear even when no sample is injected (and also appear among the genuine peaks during a sample run).

1. Peaks appear during a blank run:

   These are ghost peaks, usually found during temperature•programmedruns; the cause is contaminants trapped at the head of the column at the relatively cool starting temperature. These are released and chromatographed as column temperature rises.

   Ghost peaks are often observed when a column has been at the starting temperature for some time. For example, the first few runs in the morning often contain ghost peaks.

   - Ghost peaks may arise from septum bleed, carrier gas impurities, and contamination in plumbing by oils, grease, and other materials. Less commonly, they may be caused by reaction of stationary phase with trace levels of O_2, H_2O, and/or other materials present in the carrier gas.

- A contaminated inlet may also produce ghost peaks. Residues in the inlet are volatilized or pyrolyzed and swept onto the head of the column. Try reducing inlet temperature; if this eliminates or reduces ghosts, the inlet should be cleaned.

2. Additional peaks appear when pure sample is injected:

- These might be ghost peaks as described above. Make a blank run; if the peaks persist, they are not sample related.
- A common cause of extra peaks, assuming the sample is pure, is degradation of one or more components by an overheated inlet. Test this by reducing inlet temperature.

  Operate the inlet at as low a temperature as possible without causing peak broadening due to slow vaporization. Also, perhaps a more volatile solvent can be used. In extreme cases, derivatize the sample before analysis.
- Metal columns may also degrade the sample. Extra peaks in this case are usually broader than their immediate neighbors since they are generated along the entire length of the column. If this is the cause, changing to an all•glasssystem may be necessary.

Deformed peaks

The ideal peak, rarely occurring in chromatography, is a pure Gaussian shape. In practice, some asymmetry is always present, particularly near the baseline.

1. The peak rises normally, then drops sharply to baseline:

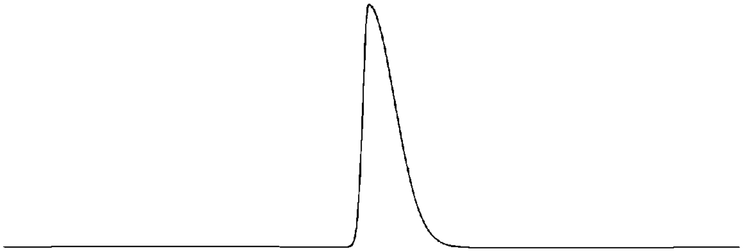
Figure 9-1.

Overloaded Peak

- The most likely cause is column overload; dilute the sample ten•foldand run it again.
- This may also be two (or more) closely merged (unresolved) peaks; lower oven temperature 30°C and repeat the analysis. If partial separation is seen, merged peaks are present.

2. The peak rises sharply and then falls normally to baseline:

Figure 9-2.

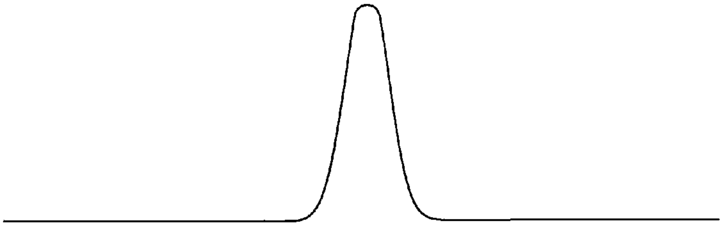


Abnormal Interaction with Column Material

- Interaction with column material is a frequent cause. Silanized support may help. An all•glasssystem may be required if metal column tubing is the source.
- Column overload with a gas sample often shows this effect; try injecting less.
- This may be a merged peak situation: Running at lower (30°C) oven temperature will increase resolution, perhaps enough to reveal merged peaks.
- Low inlet temperature may cause this, as can poor injection technique.

3. Top (apex) of the peak is deformed:

Figure 9-3.



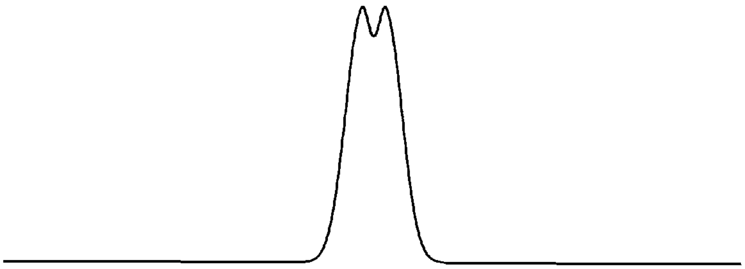
Detector Overload

- Detector overload is the probable cause: The distortion is not easily seen on the chart but reveals itself in an integrator report. In extreme cases, doubling amount injected causes little or no increase in peak size.

  Inject less sample, or dilute it, whichever is more convenient (dilution is usually the best approach). Since the detector is at the upper limit of its response, substantial dilution (100 times or more) is needed to be well within normal operating range of the detector.

4. Top (apex) of the peak is split:

Figure 9-4.



FID/NPD Flameout, or TCD with H_2 (in He Carrier)

- Verify that this is not a merged peak situation: Reduce oven temperature 30°C and repeat the run. If the **split** peak becomes better resolved, it is probably a merged pair.
- Gross overload of an FID may cause the top of the peak to invert, giving appearance of a split peak. Check gas flows; overload is more likely when flows are too low.

  Dilute the sample by a factor of at least 10 and repeat the run:
  If the split disappears, overload is the problem. It is advisable to dilute samples even more, by 100 or 1000, to ensure the detector is not close to its overload condition. Such dilution generally improves linearity as well.
- H_2 peaks, analyzed with a TCD using He carrier, often shows a split top.

Troubleshooting valve systems

Chromatographic symptoms

Troubleshooting valves and their related plumbing is primarily a matter of systematic checking and verification of unimpaired mechanical operation of any moving part. This requires an understanding of how the valve functions internally and how the plumbing is configured. A plumbing diagram is essential for effective troubleshooting.

The following symptom•causelist gives the most commonly encountered problems and solutions found with valves.

Loss of sensitivity or excessive drift

Several possible causes exist for overall deterioration of the chromatogram.

- Contamination in the valve requires a thorough cleaning.
- Internal leakage necessitates a complete disassembly and inspection of the mating surfaces.
- Poor temperature control may require a full check of electronic and thermal components.
- Lack of proper conditioning techniques, columns, etc.
- Failure or deterioration of other components (i.e., columns, detectors, etc.).

Loss of peaks in specific areas of the chromatogram

Entire sections of chromatographic data can be lost due to a valve that does not rotate or one that rotates improperly. Other than obvious component failures (i.e., solenoid, actuator, etc.), generally improper adjustments and misalignments cause most problems.

- Check that adequate air (about 482 kPa or 70 psi) is supplied.
- Check if the valve is rotating at all.
- If the valve rotates, check for proper alignment of the actuator or mechanical binding or slippage of connecting parts.
- Check for blocked flow paths with valve in both positions.

Baseline upsets

Frequently baseline upsets may be seen on chromatograms when valves are switched. These upsets are normally caused by pressure changes within the system, injections of large volume samples, or by changing the amount of restriction in the flow path. These upsets will become more of a problem when high sensitivity is required. Addition of a fixed restriction downstream from the valve may help minimize the upset. When possible, changes in column length may also help reduce the upsets. Fixed restrictors are used immediately before flame detectors to prevent flameout and are used in some instances to prevent pressure surges from damaging TCD filaments. Needle valves (Nupro) can be used as adjustable restrictors; however, they are used typically where a matched restriction is desired and not for preventing pressure or flow surges.

Often confused with baseline upsets, an offset is a shift in the baseline that does not return quickly to the original level. Baseline offsets may be caused by air leaks but more commonly are due to a change in gas purity or flow rate in the detector. Poor carrier gas or improperly conditioned filters and traps should be suspected whenever offsets occur.

Extraneous peaks

Air peaks are sometimes seen in a chromatogram when leakage occurs because the valve rotor does not seal properly. These leaks may not be detectable by using the soap•bubblemethod. The leak test procedure is described in the *Site Prep and Installation Manual*.

If a leak is suspected but cannot be located with soap bubbles, a pressure check will determine definitely if a leak exists. Extraneous peaks can occur sometimes due to improper conditioning of the valve or contamination. If leaks are not apparent, clean or condition the valve.

Obviously other causes, totally unrelated to the valve, may exhibit similar symptoms. Impure (i.e., containing water) carrier gas can cause extraneous peaks.

Locating leaks

Leak•checkingthe plumbing involved in a valve configured system must be done carefully and methodically. Several methods may be used, and the best choice will depend upon expediency, accessibility, and the magnitude of the leak. Refer to *Setting initial supply pressures and leak•testing*in the *Site Prep and Installation Manual* for details.

Pressure check

The pressure•checkmethod will indicate, but sometimes not isolate, a leak in the flow path. Since this method does not necessarily isolate the leak, one of the leak•checkmethods may be needed to locate the leak specifically.

Note that each valve in a system has two flow paths, **on** and **off**. A leak sometimes occurs in only one of these two positions. Check both.

1. Disconnect the detector from the valve system.
2. Cap the valve system at its outlet and pressurize to 689 kPa (100 psi). Allow 2 to 5 minutes for pressure to equilibrate. (If a flow sensor exists, it should read zero flow.)
3. Turn the knob on the regulator counterclockwise until it turns freely. The regulator is now turned off and the gauge is indicating pressure within the valve system.
4. Commonly, the pressure will drop quickly for approximately 30-60 seconds; then stabilize. After this initial drop, the gauge should not show more than a 6.89 to 13.78 kPa (1 to 2 psi) drop during a 10•minuteperiod.
5. If no leak is indicated, actuate all valves and repeat steps 2 through 4.
6. If a leak does show up, try to pinpoint the source with a soap•bubble technique. Do not assume the leak must exist only at a valve. Often plumbing connections such as unions or bulkhead fittings are at fault.

7. If the leak cannot be found easily, divide the system in half and repeat the pressure check. Continue dividing by halves, and pressure check until the leak is isolated.

Electronic pressure control

The electronic pressure control option provides very accurate and precise control of column head pressure, resulting in retention time reproducibility of better than 0.02% RSD when there are no column effects. The inlet pressure can be set constant, programmed, or set to maintain a desired column flow rate. This mass flow control can be maintained even at vacuum column outlet pressures. A safety shutdown feature stops the run and can trigger an alarm if the column breaks or pressure otherwise falls. In addition, a bypass flow maintains a small positive column pressure in the event of power failure, protecting the analytical column.

Electronic Pressure Control Problems

Symptom	Possible Cause
Not enough pressure (Safety shutdown activated)	1. Septum leaks or is missing 2. Column is broken 3. Column ferrule seal leaks 4. Gas supply is off 5. Supply pressure is inadequate 6. Desired pressure may not be achieveable with the column in use.
Pressure goes to O or max.	1. Configuration is wrong. See the section *Proper configuration.*
Not Ready light flickers (oscillating pressure)	1. Septum and/or column connection leaks. 2. Pressure set higher than the operating limit
Pressure not controllable	1. Configuration is wrong. See the section *Proper configuration.*

If you have checked these possible causes and still have a problem, call HP Service.

Safety shutdown

Systems equipped with electronic pressure programming have a safety shutdown feature to prevent gas leaks from creating a safety hazard. If the system cannot reach a pressure setpoint it beeps. After about 45 seconds the beep will stop and the message:

EPPB: SAFETY SHUTDOWN

will appear on the display, and the system will shut down by turning **off** all electronic pressure and heated zones, and locking the keyboard.

A safety shutdown can occur when:

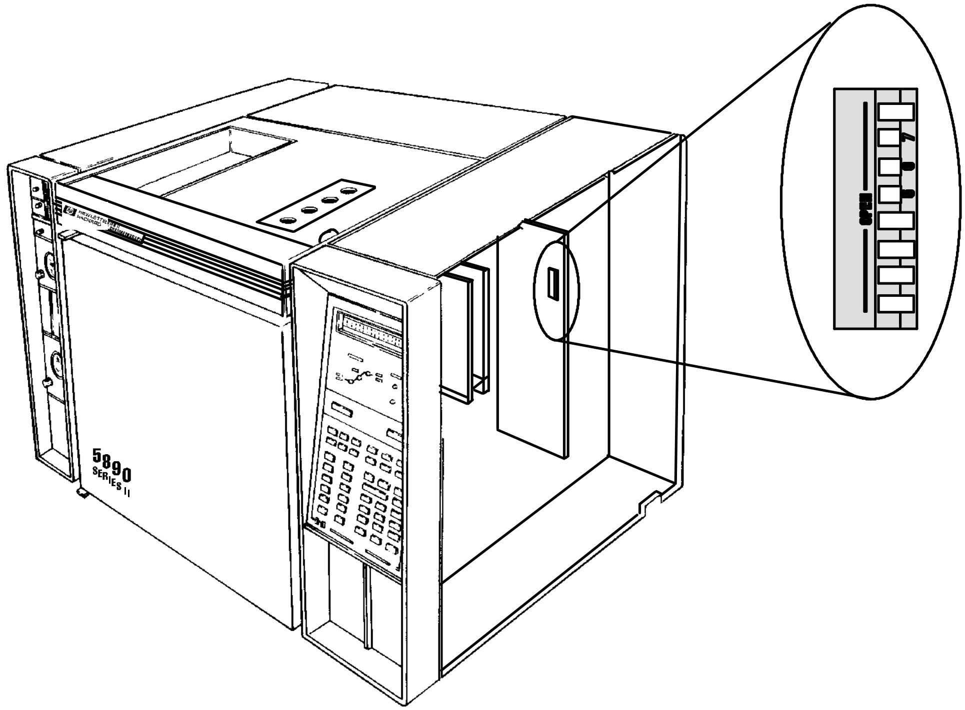
1. There is a leak in the system (see *Pressure control problems*). This includes missing septa or columns!
2. The column is not restrictive enough to reach desired pressure (i.e., 530 µ columns will not go to 100 psi with available flow). Note: When using pressure programming, this may occur during a pressure ramp to too high a pressure.
3. There is insufficient supply pressure.
4. Configuration is set wrong. Check the mode switch on the inlet controller board (see *Proper configuration*).

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

Proper configuration

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

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

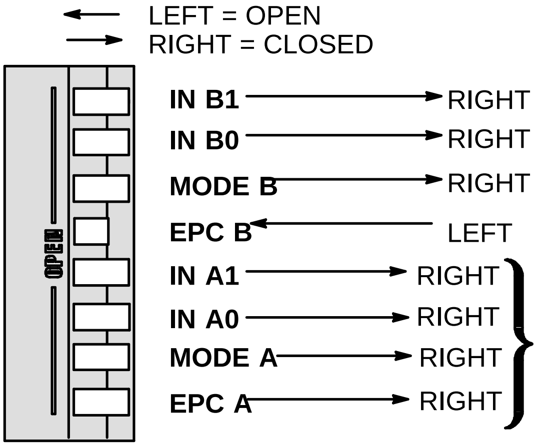


Switch setting examples

IN A1 or IN B1	RIGHT, currently unused PID
IN A0 or IN B0	LEFT, Programmable Cool On-Column (PID) / RIGHT, Purged Packed Inlet & Split/Splitless Capillary Inlet (PID)
MODE A or MODE B	LEFT, (FPR) Programmable Cool On-Column Inlet & Purged Packed Inlet / RIGHT, (BPR) Split/Splitless Capillary Inlet
EPC A or EPC B	LEFT, Electronic Pressure Control present / RIGHT, Electronic Pressure Control not present

EXAMPLE

INLET B = Split/Splitless Inlet with Electronic Pressure Control
INLET A = Any Non-Electronic Pressure Controlled Inlet



NOTE

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

10

Test Sample Chromatograms

Test Sample Chromatograms

This chapter contains typical examples of test sample chromatograms. They may be used as a general guide to instrument performance.

It is assumed that both the instrument and proper test column are installed, that general keyboard control is understood (temperature control, defining signal output, etc.), and that specific operating information for the given inlet and detector is also understood. If not, consult the *HP 5890 SERIES II Operating Manual* as necessary before proceeding with test chromatogram procedures.

Note that injection volumes listed with operating conditions in the following chromatograms do not necessarily indicate total absolute volume injected. Volume given is simply the graduation (plunger position) read from a standard 10 µl syringe. For a heated inlet, actual sample volume injected will also include an additional 0.4-0.7 µl, the volume of sample volatilized from inside the syringe needle. For the dedicated on•columninlet (unheated), the syringe plunger position more accurately represents true injected volume.

Also note that the following procedures and results are intended only to provide evidence of a properly functioning inlet and/or detector system; they are not necessarily suitable to test a given system against its specification limits.

Test sample chromatograms

Figure 10-1.

HP 5890 Test Sample Operating Conditions

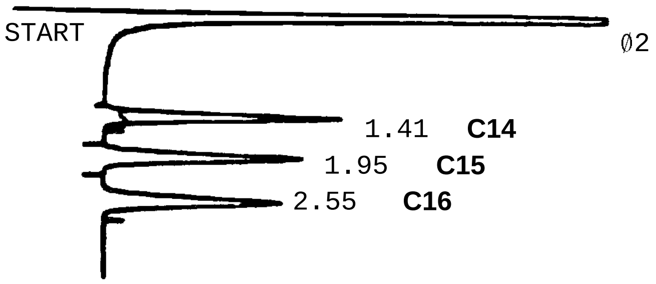
Detector Type FID (or FIDw/MUG)
Temp 250 DEGREES C
Inlet Type PACKED (OR PURGED PACKED).
Temp 200 DEGREES C
Operating Mode N/A
Purge Time On N/A min
Purge Time Off N/A min
Oven Temp Programmed (1 ramp)
Init Temp 110 DEGREES C
Init Time 0 min
Ramp
Rate 15
Fin Temp 150
Fin Time 1
Range 8

COLUMN:
Part No. 19095(#100)
Dimensions 530µ MID; 5 M
Sta Phase Methyl Silicone

FLOW RATES

Carrier (He)	20 +/• 1	ml/min
Hydrogen	33 +/• 1	ml/min
Air	400 +/• 20	ml/min
Makeup (N2)	10 +/• 1	ml/min
Split Vent	N/A	ml/min
Septum Purge	1•2	ml/min

SAMPLE:
Type FID Sample
Inj Volume 1 µl
Part No. 18710•60170
Composition 0.03%(V/V)each C14,C15,C16 normal paraffin hydrocarbons in hexane

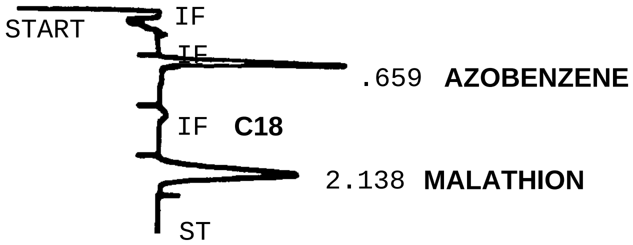


Flame Ionization Detector (FID)

Figure 10-2.

HP 5890 Test Sample Operating Conditions

```
Detector Type NPD (or NPDw/MUG)  FLOW RATES
         Temp 220 DEGREES C      Carrier (He)  20 +/• 1    ml/min
Inlet Type PACKED (OR PURGED     Hydrogen    3.5 +/• 0.1   ml/min
            PACKED).             Air       100•120 +/• 10 ml/min
         Temp 170 DEGREES C      Makeup (He)    10+/•1     ml/min
Operating Mode    N/A            Split Vent      N/A       ml/min
Purge Time On     N/A      min   Septum Purge    1•2       ml/min
Purge Time Off    N/A      min
Oven Isothermal
   Init Temp    170  DEGREES C
   Init Time    3.0    min
   Ramp                          SAMPLE:
   Rate           0              Type   NP-FID Sample
   Fin Temp     ___              Inj Volume   3 µl
   Fin Time     ___              Part No. 18789•60060
Range 0                          Composition 0.65 ppm azobenzene,
                                  100 ppm octadecane, 1.0 ppm
COLUMN:                           malathion in isooctane
Part No.    19095S(#100)
Dimensions     530µ MID; 5 M
Sta Phase   Methyl Silicone
```



Nitrogen-Phosphorus Detector (NPD)

Figure 10-3.

HP 5890 Test Sample Operating Conditions

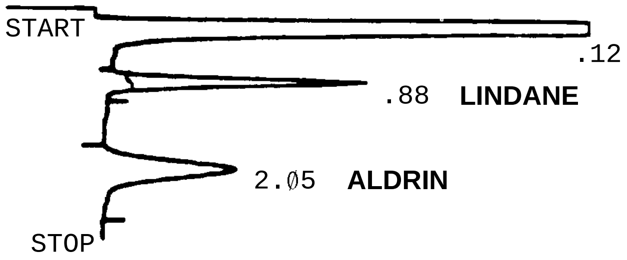
Detector Type ECD(or ECDw/MUG)
Temp 300 DEGREES C
Inlet Type PACKED (OR PURGED PACKED).
Temp 200 DEGREES C
Operating Mode N/A
Purge Time On N/A min
Purge Time Off N/A min
Oven Temp Isothermal
Init Temp 160 DEGREES C
Init Time N/A min
Ramp
Rate 0
Fin Temp ____
Fin Time ____
Range 2

COLUMN:
Part No. 19095S(#100)
Dimensions 530µ MID; 5 m
Sta Phase Methyl Silicone

FLOW RATES

Carrier (N2)	30 +/• 1	ml/min
Hydrogen	N/A	ml/min
Air	N/A	ml/min
Makeup (N2)	30 +/• 1	ml/min
Split Vent	N/A	ml/min
Septum Purge	1•2	ml/min

SAMPLE:
Type ECD Sample
Inj Volume 1 µl
Part No. 18713•60040
Composition 33 pg/µl(0.033 ppm(W/V)) each: and aldrin in isooctane

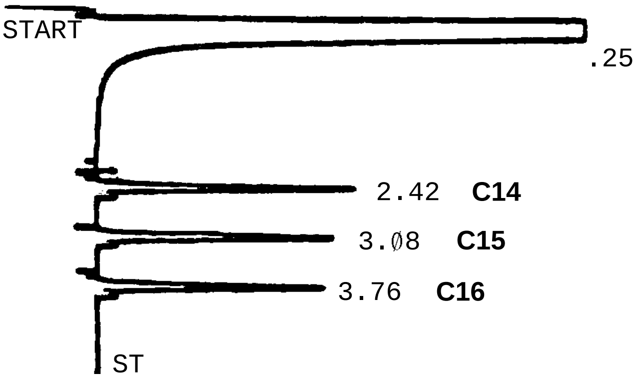


Electron Capture Detector (ECD)

Figure 10-4.

HP 5890 Test Sample Operating Conditions

```
Detector Type TCD(or TCDw/MUG)     FLOW RATES
         Temp 300 DEGREES C        Carrier (He)  20 +/• 1    ml/min
Inlet Type PACKED (OR PURGED       Hydrogen         N/A      ml/min
            PACKED).               Air              N/A      ml/min
         Temp 250 DEGREES C        Makeup (He)      1•2      ml/min
Operating Mode    N/A              Split Vent       N/A      ml/min
Purge Time On     N/A      min     Septum Purge     1•2      ml/min
Purge Time Off    N/A      min     Reference (He) 30+/•1     ml/min
Oven Temp Programmed (1 ramp)
   Init Temp    110  DEGREES C
   Init Time      1    min
   Ramp
   Rate        15                  SAMPLE:
   Fin Temp    150                 Type   FID Sample
   Fin Time    1                   Inj Volume   3 µl
Range    0                         Part No. 18710•60170
                                   Composition 0.03%(V/V)each
COLUMN:                              C14,C15,C16 normal paraffin
Part No.    19095S(#100)             hydrocarbons in hexane
Dimensions     530µ MID; 5 M
Sta Phase   Methyl Silicone
```



Thermal Conductivity Detector (TCD)

Figure 10-5.

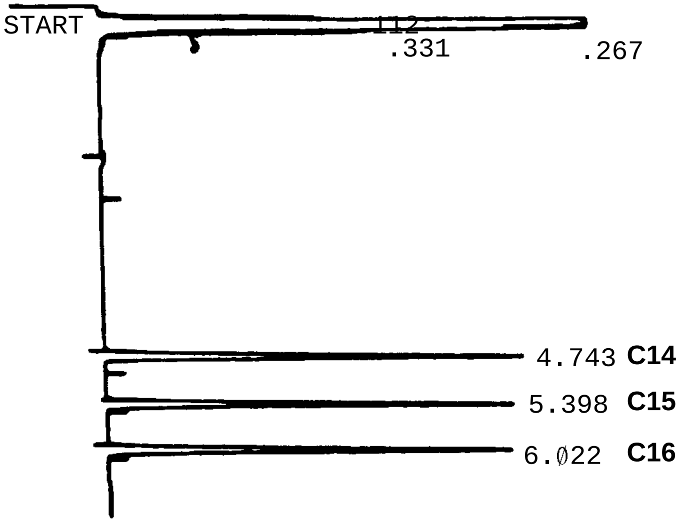
HP 5890 Test Sample Operating Conditions

```
Detector Type  FIDw/MUG
         Temp 250 DEGREES C
Inlet Type Ded On-Col Cap
Oven Track        On
         Temp N/A          C
Operating Mode    N/A
Purge Time On     N/A      min
Purge Time Off    N/A      min
Oven Temp Temp Programmed (1
ramp)
   Init Temp     60 DEGREES C
   Init Time    0.5   min
   Ramp
   Rate        20
   Fin Temp    185
   Fin Time    1
Flow Param (EPP)
   Constant Flow    Off
Range    4

COLUMN:
Part No.    19095(#100)
Dimensions     530µ MID; 5 M
Sta Phase   Methyl Silicone
```

```
FLOW RATES
Carrier (He)  15 +/• 1     ml/min
Hydrogen      30 +/• 1     ml/min
Air          400 +/• 20    ml/min
Makeup (N2)   20 +/• 1     ml/min
Split Vent      N/A        ml/min
Septum Purge   5 +/• 1     ml/min
```

```
SAMPLE:
Type   FID Sample
Inj Volume   1 µl
Part No. 18710•60170
Composition 0.03%(V/V)each:
C14,C15, and C16 normal paraffin
hydrocarbons in hexane.
```



FID-On-Column Capillary Inlet

Figure 10-6.

HP 5890 Test Sample Operating Conditions

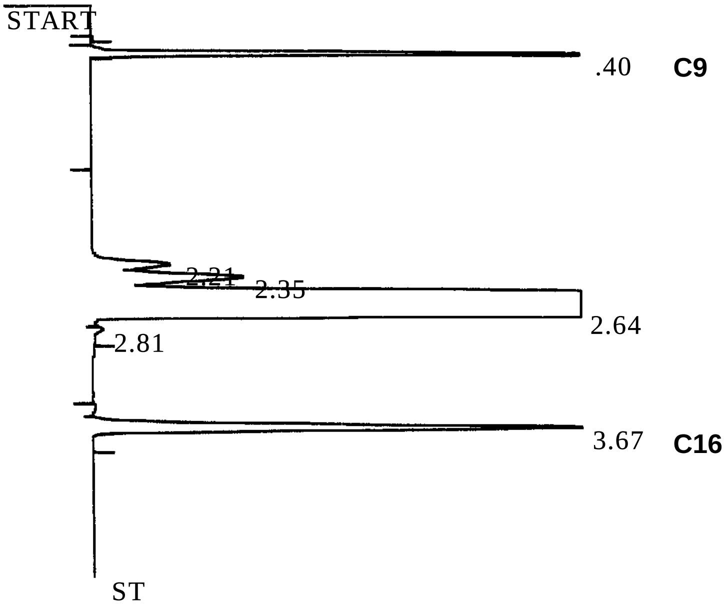
Detector Type FIDw/MUG
Temp 250 DEGREES C
Inlet Type SPLIT ONLY OR
SPLIT/SPLITLESS Temp 200 DEG C
Operating Mode SPLIT(PURGE ON)
Purge Time On 0 min
Purge Time Off 0 min
Oven Temp Programmed (1 ramp)
Init Temp 100 DEGREES C
Init Time 0 min
Ramp
Rate 20
Fin Temp 180
Fin Time 1
Range 7

COLUMN:
Part No. 19095Z•121
Dimensions 530µ ; 10 M
Sta Phase Methyl Silicone

FLOW RATES

Carrier (He)	15 +/• 1	ml/min
Hydrogen	30 +/• 1	ml/min
Air	400 +/• 20	ml/min
Makeup (N2)	20 +/• 1	ml/min
Split Vent	200 +/• 20	ml/min
Septum Purge	5 +/• 1	ml/min

SAMPLE:
Type Capillary Sample
Inj Volume 1 µl
Part No. 18740•60900
Composition: Soln of 0.1%(w/w) n-nonane,n-hexadecane and 0.1% (w/w)each of 1-octanol,nonanol, n-undecane,naphthalene,2-decanone,n-dadecane,n-tridecane, 1-pentadecane in tetradecane.



FID-Split Mode Capillary Inlet

Figure 10-7.

HP 5890 Test Sample Operating Conditions

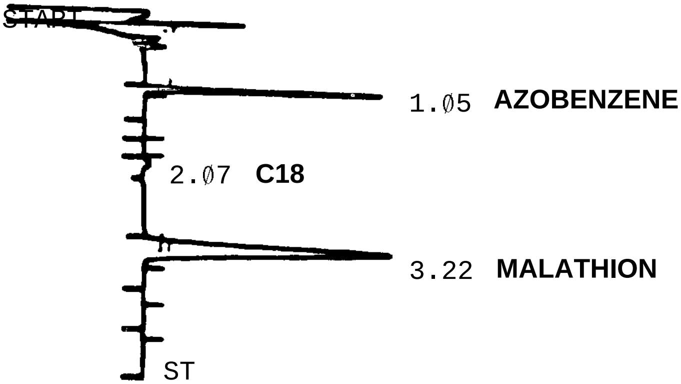
Detector Type NPD w/MUG
Temp 220 DEGREES C
Inlet Type Split only or split/splitless
Temp 200 DEGREES C
Operating Mode Split(Purge on)
Purge Time On 0 min
Purge Time Off 0 min
Oven Isothermal
Init Temp 180 DEGREES C
Init Time 5.0 min
Ramp
Rate 0
Fin Temp ____
Fin Time ____
Range 0

COLUMN:
Part No. 19095Z(#121)
Dimensions 530µ MID; 10 M
Sta Phase

FLOW RATES

Carrier (He)	15 +/• 1	ml/min
Hydrogen	3.5 +/• 0.1	ml/min
Air	100•120 +/• 10	ml/min
Makeup (He)	10+/•1	ml/min
Split Vent	80+/•5	ml/min
Septum Purge	5+/•1	ml/min

SAMPLE:
Type NP-FID Sample
Inj Volume 2 µl
Part No. 18789•60060
Composition 0.65 ppm azobenzene, 100 ppm octadecane, 1.0 ppm malathion in isooctane



NPD-Split Mode Capillary Inlet

Figure 10-8.

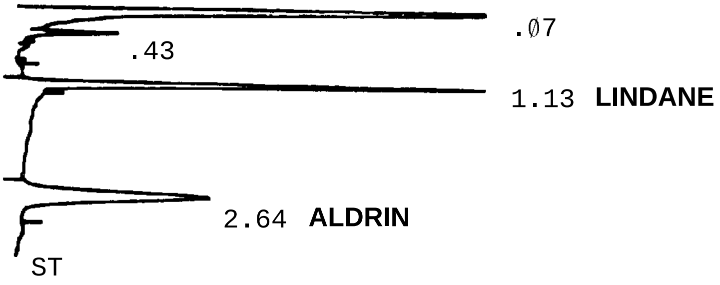
HP 5890 Test Sample Operating Conditions

Detector Type ECDw/MUG
Temp 300 DEGREES C
Inlet Type Split only or splitless
Temp 200 DEGREES C
Operating Mode Split(Purge on)
Purge Time On 0 min
Purge Time Off 0 min
Oven Temp Isothermal
Init Temp 170 DEGREES C
Init Time N/A min
Ramp
Rate 0
Fin Temp ____
Fin Time ____
Range 0

COLUMN:
Part No. 19095S(#100)
Dimensions 530µ MID; 5 m
Sta Phase Methyl Silicone

FLOW RATES		
Carrier (He)	15 +/• 1	ml/min
Hydrogen	N/A	ml/min
Air	N/A	ml/min
Makeup (N2)	60 +/• 2	ml/min
Split Vent	60 +/• 2	ml/min
Septum Purge	5 +/• 1	ml/min

SAMPLE:
Type ECD Sample
Inj Volume 1 µl
Part No. 18713•60040
Composition 33 pg/µl (0.033 ppm (W/V)) each: lindane and aldrin in isooctane



ECD-Split Mode Capillary Inlet

Figure 10-9.

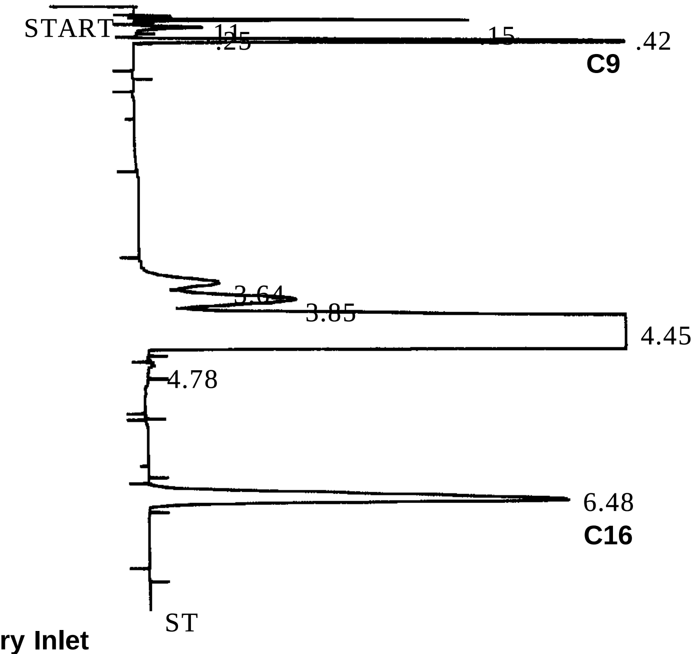
HP 5890 Test Sample Operating Conditions

Detector Type TCDw/MUG
Temp 300 DEGREES C
Inlet Type Split only or split/splitless
Temp 250 DEG C
Operating Mode Split(Purge on)
Purge Time On 0 min
Purge Time Off 0 min
Oven Temp Programmed
Init Temp 100 DEGREES C
Init Time 1 min
Ramp
Rate 10
Fin Temp 150
Fin Time 2
Range 0

COLUMN:
Part No. 19095Z(121)
Dimensions 530µ MID; 5 M
Sta Phase Methyl Silicone

FLOW RATES		
Carrier (He)	15 +/• 1	ml/min
Hydrogen	N/A	ml/min
Air	N/A4	ml/min
Makeup (He)	10 +/• 1	ml/min
Split Vent	200 +/• 10	ml/min
Septum Purge	5 +/• 1	ml/min
Reference (He)	37 +/• 1	ml/min

SAMPLE:
Type Capillary
Inj Volume 1 µl
Part No. 18740•60900
Composition See Split/FID



TCD-Split Mode Capillary Inlet

Figure 10-10.

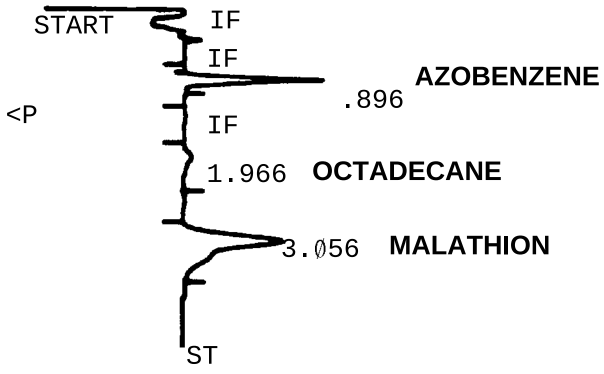
HP 5890 Test Sample Operating Conditions

Detector Type NPD w/MUG)
Temp 220 DEGREES C
Inlet Type Ded On-Col Cap
Oven Track On
Temp N/A C
Operating Mode N/A
Purge Time On _____ min
Purge Time Off _____ min
Oven Isothermal
  Init Temp 170 DEGREES C
  Init Time 5.0 min
  Ramp
  Rate 0
  Fin Temp ____
  Fin Time ____
Flow Param (EPP)
  Constant Flow Off
Range 0

FLOW RATES		
Carrier (He)	15 +/• 1	ml/min
Hydrogen	4 +/• 1	ml/min
Air	90 +/• 10	ml/min
Makeup (He)	10+/•1	ml/min
Split Vent	N/A	ml/min
Septum Purge	5+/•1	ml/min

SAMPLE:
Type NP-FID Sample
Inj Volume 3 µl
Part No. 18789•60060
Composition 0.65 ppm azobenzene, 100 ppm octadecane, 1.0 ppm malathion in isoioctane

COLUMN:
Part No. 19095S(#100)
Dimensions 530µ MID; 5 M
Sta Phase Methyl Silicone

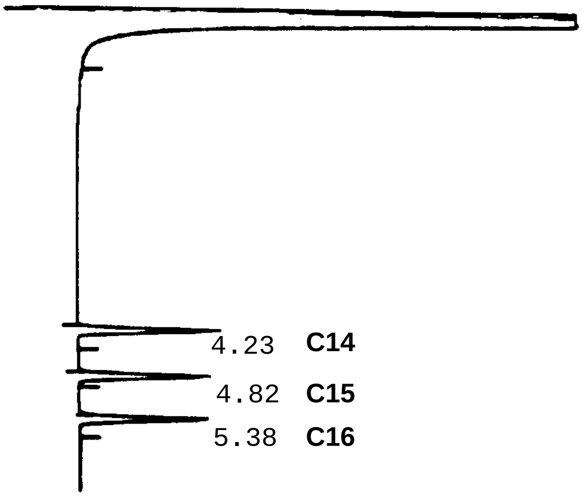


NPD-On-Column Capillary Inlet

Figure 10-11.

HP 5890 Test Sample Operating Conditions

```
Detector Type  TCDw/MUG             FLOW RATES
         Temp 300 DEGREES C         Carrier (He)  20 +/• 1     ml/min
Inlet Type Ded On-Col               Hydrogen        N/A        ml/min
Oven Track        On                Air             N/A        ml/min
         Temp N/A          C        Makeup (He)    5+/•1       ml/min
Operating Mode    N/A               Split Vent      N/A        ml/min
Purge Time On     N/A      min      Septum Purge   5+/•1       ml/min
Purge Time Off    N/A      min      Reference (He) 37+/•1      ml/min
Oven Temp Programmed (1 ramp)
    Init Temp     60  DEGREES C
    Init Time    0.5    min
    Ramp
    Rate        20                  SAMPLE:
    Fin Temp    180                 Type   FID Sample
    Fin Time    1                   Inj Volume   3 µl
Flow Param (EPP)                    Part No. 18710•60170
    Constant Flow     Off           Composition 0.03%(V/V)each
Range    0                           C14,C15,C16 normal paraffin
                                     hydrocarbons in hexane.
COLUMN:
Part No.    19095S(#100)
Dimensions     530µ MID; 5 M
Sta Phase   Methyl Silicone
```



TCD-On-Column Capillary Inlet

Figure 10-12.

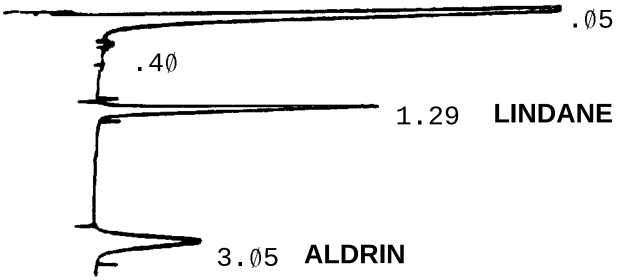
HP 5890 Test Sample Operating Conditions

Detector Type ECDw/MUG
Temp 300 DEGREES C
Inlet Type Ded On-Column
Oven Track On
Temp N/A
Operating Mode N/A
Purge Time On N/A min
Purge Time Off N/A min
Oven Isothermal
Init Temp 170 DEGREES C
Init Time N/A min
Ramp
Rate 0
Fin Temp
Fin Time
Flow Param (EPP)
Constant Flow Off
Range 2

COLUMN:
Part No. 19095S(#100)
Dimensions 530µ MID; 5 m
Sta Phase Methyl Silicone

FLOW RATES		
Carrier (He)	15 +/• 1	ml/min
Hydrogen	N/A	ml/min
Air	N/A	ml/min
Makeup (N2)	60+/•2	ml/min
Split Vent	N/A	ml/min
Septum Purge	60+/•1	ml/min

SAMPLE:
Type ECD Sample
Inj Volume 1 µl
Part No. 18713•60040
Composition 33 pg/µl
(0.033 ppm (W/V)) each: lindane and aldrin in isooctane



ECD-On-Column Capillary Inlet

Figure 10-13.

HP 5890 Test Sample Operating Conditions

Detector Type FPD
Temp 200 DEGREES C
Inlet Type PACKED OR PURGED PACKED
Temp 200 DEGREES C
Operating Mode N/A
Purge Time On N/A min
Purge Time Off N/A min
Oven Temp Programmed (1 ramp)

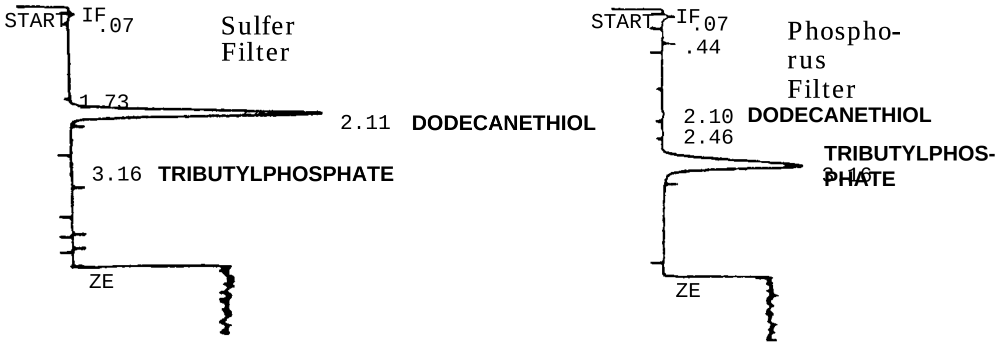
Init Temp	110	DEGREES C
Init Time	0	min
Ramp Rate	10	
Fin Temp	170	
Fin Time	3	

Range 5
COLUMN:
Part No. 19095S•100
Dimensions 5 M Length, 530 µ ID
Sta Phase Methyl Silicone

FLOW RATES (FPD AIR MODE FLOWS)

Carrier (N2)	20 +/• 1	ml/min
Hydrogen	75 +/• 2	ml/min
Air	100 +/• 3	ml/min
Makeup (N2)	0	ml/min
Split Vent	N/A	ml/min
Septum Purge	1•2	ml/min

SAMPLE:
Type FPD Sample
Inj Volume 1 µl
Part No. 19395•60580
COMPOSITION: 20 ng/µl
(20.0 ppm W/V) each of
1-dodecanethiol and
tributylphosphate in isooctane



FPD Packed Column Inlet

Figure 10-14.

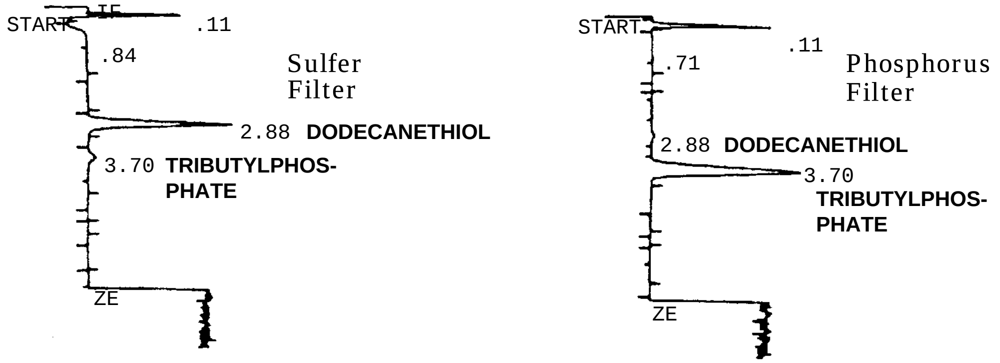
HP 5890 Test Sample Operating Conditions

Detector Type FPD
Temp 200 DEGREES C
Inlet Type SPLIT ONLY OR SPLIT/SPLITLESS
Temp 200 DEGREES C
Operating Mode SPLIT(PURGE ON)
Purge Time On 0 min
Purge Time Off 0 min
Oven Temp Programmed (1 ramp)
Init Temp 110 DEGREES C
Init Time 0 min
Ramp Rate 10
Fin Temp 170
Fin Time 3
Range 5
COLUMN:
Part No. 19095Z•121
Dimensions 5 M Length, 530 µ ID
Sta Phase Methyl Silicone

FLOW RATES (FPD AIR MODE FLOWS)

Carrier (N2)	20 +/• 1	ml/min
Hydrogen	75 +/• 2	ml/min
Air	100 +/• 3	ml/min
Makeup (N2)	0	ml/min
Split Vent	200 +/• 20	ml/min
Septum Purge	5 +/• 1	ml/min

SAMPLE:
Type FPD Sample
Inj Volume 2 µl
Part No. 19305•60580
COMPOSITION: 20 ng/µl (20.0 ppm W/V) each of 1-dodecanethiol and tributylphosphate in isooctane



FPD Split Mode Capillary Inlet

Figure 10-15.

HP 5890 Test Sample Operating Conditions

Detector Type FPD
Temp 200 DEGREES C
Inlet Type DED ON-COL CAP
Oven Track On
Temp N/A DEGREES C
Operating Mode N/A
Purge Time On N/A min
Purge Time Off N/A min
Oven Temp Programmed (1 ramp)

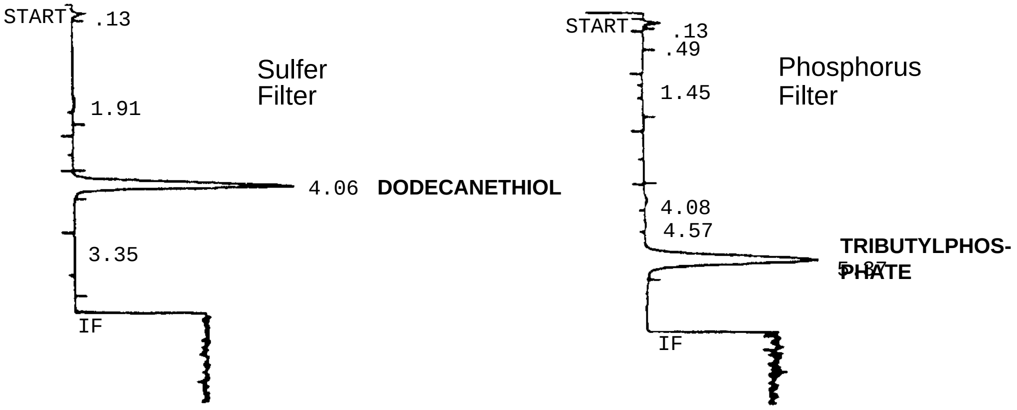
Init Temp	90	DEGREES C
Init Time	1.0	min
Ramp	(1)	(2)
Rate	20	10
Fin Temp	110	170
Fin Time	0	3

Flow Param (EPP)
Constant Flow Off
Range 5
COLUMN:
Part No. 19095S•100
Dimensions 5 M Length, 530 μ ID
Sta Phase Methyl Silicone

FLOW RATES (FPD AIR MODE FLOWS)

Carrier (N2)	20 +/• 1	ml/min
Hydrogen	75 +/• 2	ml/min
Air	100 +/• 3	ml/min
Makeup (N2)	0	ml/min
Split Vent	N/A	ml/min
Septum Purge	5 +/• 1	ml/min

SAMPLE:
Type FPD Sample
Inj Volume 1 μl
Part No. 19395•60580
COMPOSITION: 20 ng/μL
(20.0 ppm W/V) each of
1-dodecanethiol and
tributylphosphate in isooctane



FPD On-column Inlet

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