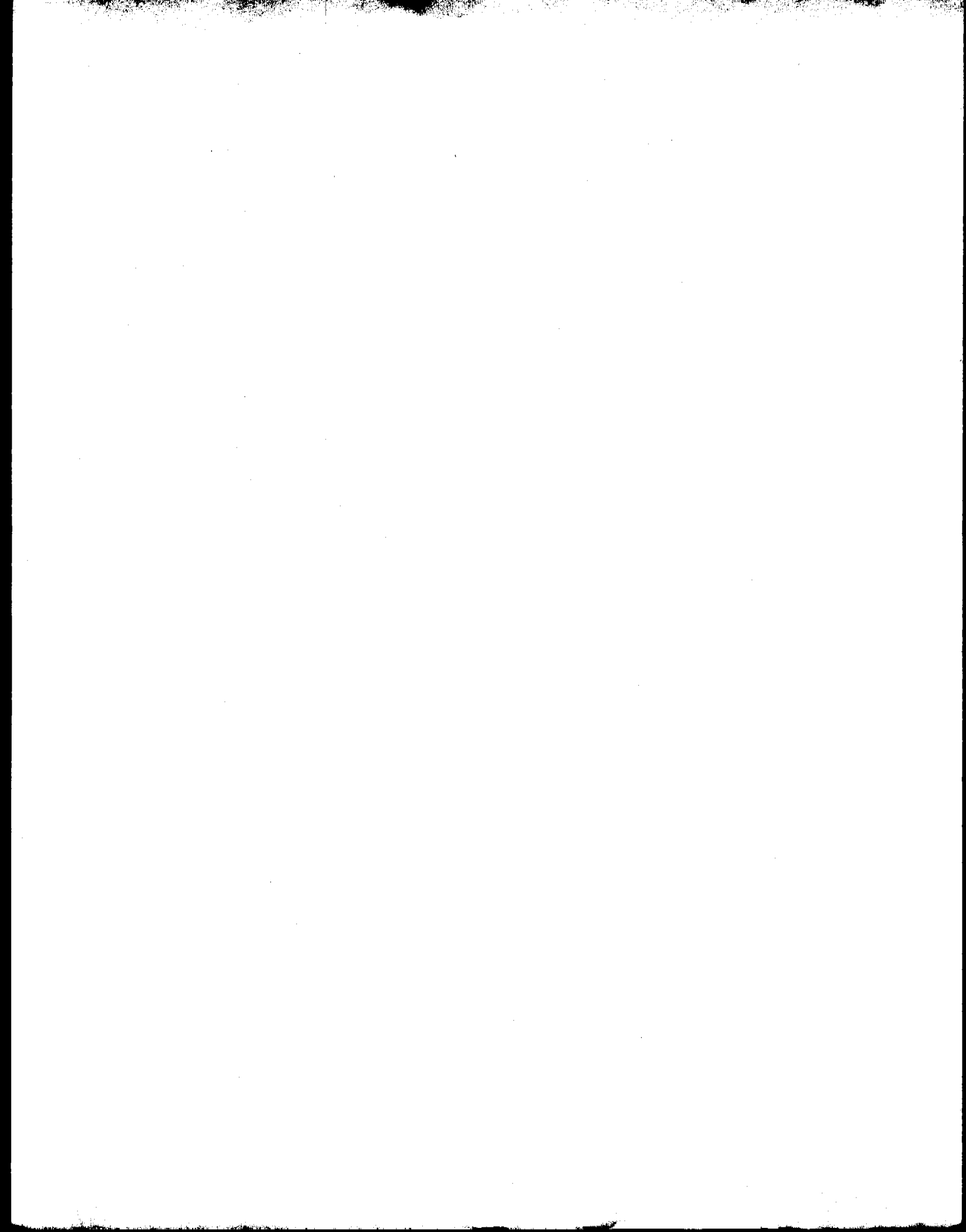


HP 8452 Automatic
Liquid Sampler

Operating Manual



Operating Manual

Automatic Liquid Sampler

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1995

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Printed in USA

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Sampler meets the following
IEC (International Electro-
technical Commission) classi-
fications: Safety Class 1, Tran-
sient Overvoltage Category II,
and Pollutions Degree 2. This
unit has been designed and
tested in accordance with
recognized safety standards
and designed for use indoors.
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tion of the HP Automatic
Liquid Sampler has been
compromised, disconnect the
unit from all power sources
and secure the unit against
unintended operation.

The recyclable carbon mono-
fluoride lithium battery is a
BR-2/3 A 1,200 mAh. Fuses
F001 and F002 are 3 A,
250 Vac, IEC 127 Type T.
Fuses F201 and F202 are
10 A, 250 Vac, IEC 127
Type T. Fuse F101 is a 0.5 A,
250 Vac.

Warnings in this manual or on
the instrument must be
observed during all phases of
operation, service, and repair
of this instruments. Failure to
comply with these precautions
violates safety standards of
design and the intended use
of the instrument. Hewlett-
Packard assumes no liability
for the customer's failure to

comply with these require-
ments.

Refer servicing to qualified
service personnel. Substituting
parts or performing any
unauthorized modification to
the instrument may result in a
safety hazard. Disconnect the
AC power cord before remov-
ing covers. The customer
should not attempt to replace
the battery or fuses in this
instrument.

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 (method-
ology) 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 $L_p < 70\text{db(A)}$
During normal operation
At the operator position
According to ISO 7779 (Type
Test)

When operating the
HP Automatic Liquid Sampler
with cryo valve option, the
sound pressure $\approx 78\text{db(A)}$
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
Maschinenlärminformationsv-
erordnung vom 18 Januar
1991.

Schalldruckpegel $L_p < 70$
dB(A)

Am Arbeitsplatz
Normaler Betrieb
Nach DIN 45635 T. 19
(Typprüfung)

Bei Betrieb des
HP Automatischer
Slüssigkeitsprobengeber mit
Cryo Ventil Option treten
beim Oeffnen des Ventils
impulsfoermig Schalldrucke
 L_p bis ca. 78 dB(A) auf.

Little Falls Site
Hewlett-Packard Company
2850 Centerville Road
Wilmington, DE 19808-1610

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Setting Up

Setting Up

The purpose of this chapter is to:

- Introduce you to the names of the major parts associated with installing the automatic liquid sampler and some preventive maintenance tips.
- Help you set up the automatic liquid sampler and turn on the power.

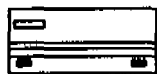
You must complete the following tasks during installation to be ready to turn on the power.

1. Install the mounting brackets for the injector and tray, along with any upgrade parts for the gas chromatograph (GC). Instructions for these installations are contained in link manuals that are packaged with the brackets or upgrade parts.
2. Mount the injector and tray on the brackets.
3. Verify that the controller switch settings are correct.
4. Connect the cables for the injector and tray to the controller.
5. Connect the communication cables for the controller, the GC, and the data handling device.
6. Install the power cord for the controller.

The instructions for tasks 2 through 6 start on page 11.

The modules of the automatic liquid sampler

The automatic liquid sampler is made up of three modules:



The HP G1512A controller and communications module supplies power and communications to the injector and tray.



The HP G1513A injector module removes a volume of sample from a vial and injects it into the inlet.



The HP 18596C tray module moves sample vials to and from the injector and the bar code reader.

The automatic liquid sampler works with the PrepStation system to automate sample preparation procedures. The HP G1296A/HP G1926A bar code reader module is also available. It reads the vial numbers and special instructions for running a method. It can also be used for agitating the sample vial.

The automatic liquid sampler becomes part of your gas chromatography system. It introduces sample to an inlet or a column on your GC.

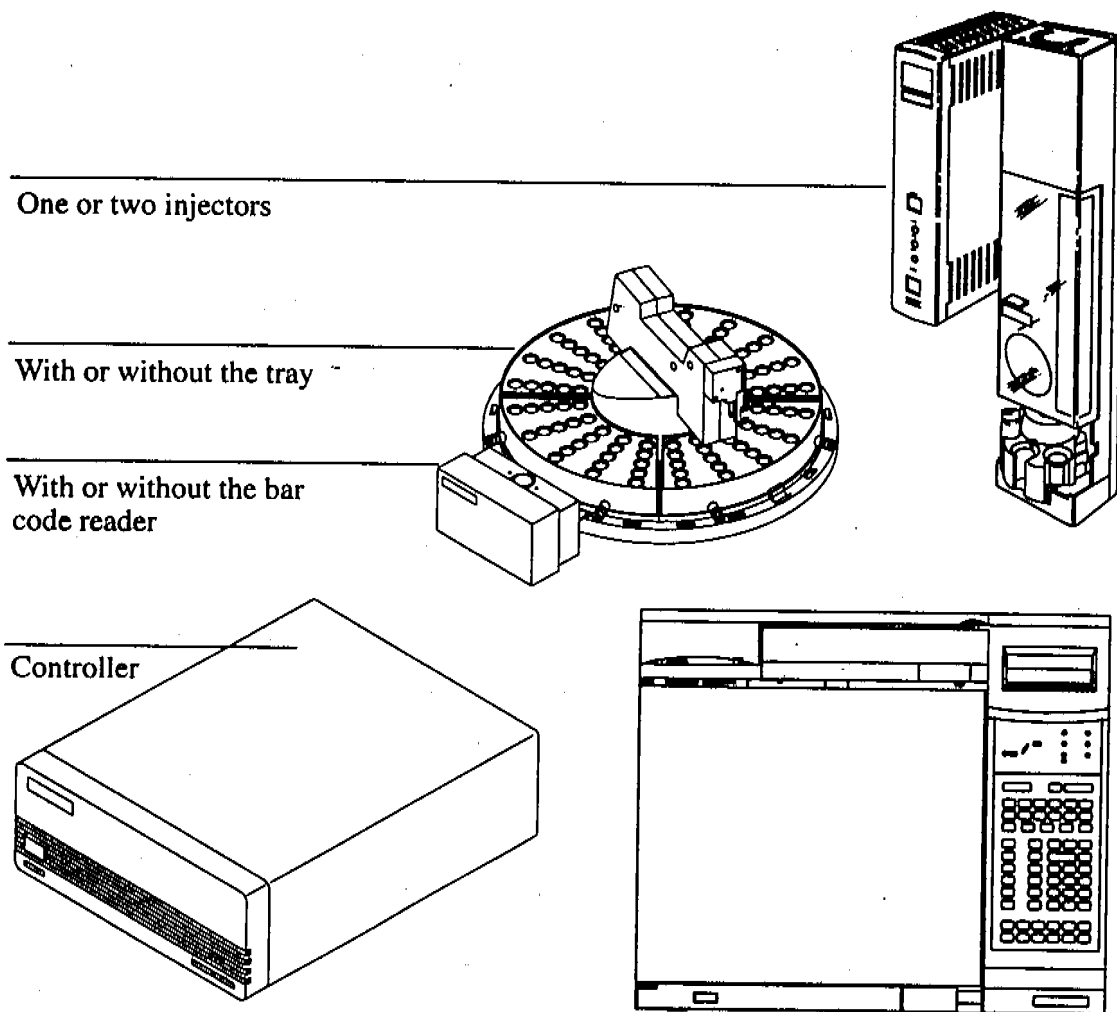
The automatic liquid sampler, with or without the tray, can be controlled by a variety of integrators and computers, including:

- HP 6890 Series GC
- Its own electronics (standalone control)
- HP 3396 integrators plus older models
- Most HP laboratory automation systems
- Most HP ChemStation systems
- Suitably programmed external computers

Setting Up
The modules of the automatic liquid sampler

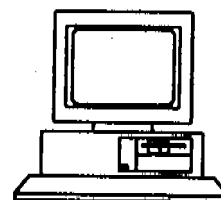
Figure 1-1 illustrates how the automatic liquid sampler is an integral part of the analytical system.

Figure 1-1



Controller configured to communicate with:

- HP 6890 Series GC
- Integrator
- Personal computer
- Workstation
- Lab data system
- Injector's control switches



The automatic liquid sampler as part of your analytical system.

Preparing the site

The automatic liquid sampler has certain environmental, space, and electrical power requirements.

Environmental considerations

The automatic liquid sampler is designed to operate within ranges of temperature and relative humidity.

- Temperature range: 0° to 55°C
- Relative humidity range: 5% to 95% at 40°C
- Altitude ranges: up to 2,300 m

Hewlett-Packard recommends an environment comfortable for the operators (reasonably constant temperature and humidity) for optimum performance and instrument lifetime.

Caution

Do not place any instruments that release heat on top of or underneath the controller. For example, do not place an integrator on top of the controller. The additional heat can cause damage to its electrical components.

Space considerations

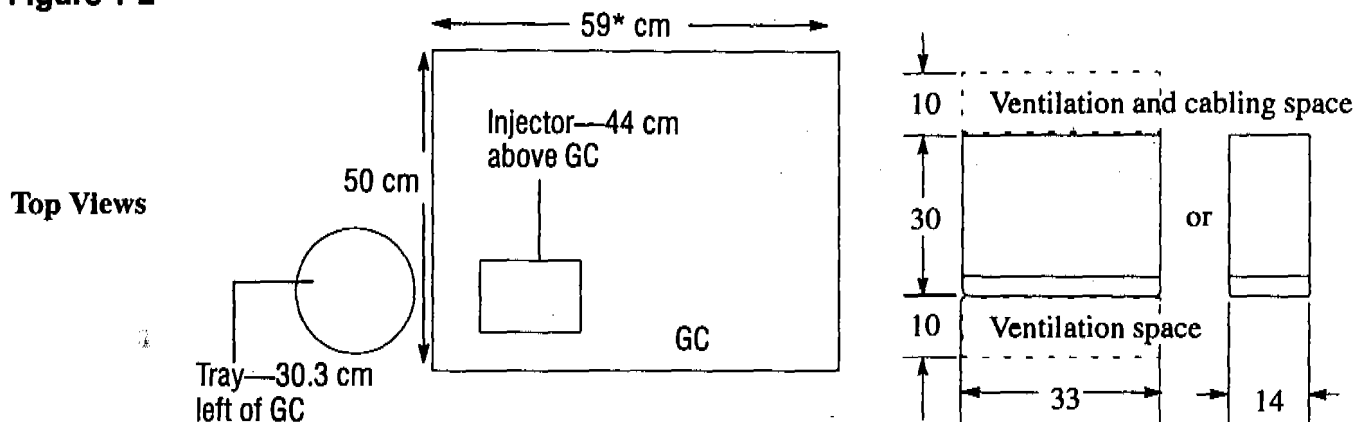
The injector and tray increase the space requirements for the GC. The exact amount depends on the model of the GC. The illustration on the following page shows the space requirements for the components with an HP 6890 Series GC.

Installed on the HP 6890 Series GC, the injector adds 44 cm (17 in.) in height. The tray adds 30.3 cm (9 in.) to the left side and 3 cm (2 in.) to the front. The controller requires a clear surface about 33 cm (13 in.) wide and 38 cm (15.2 in.) deep. About 20 cm (8 in.) of the depth is required for cabling. If you place the controller on its right-hand side (i.e., power switch up), it needs a clear surface of 38 cm (15.2 in.) deep and 14 cm (6 in.) wide.

WARNING

Place the HP G1512A controller where you can easily unplug it from the power source.

Figure 1-2



All dimensions in centimeters

*68 cm for non-EPC version.

Space considerations.

Electrical power requirements

The controller is the power source for the injector and tray. The controller has an autoranging power supply:

- 100–240 Vac single phase
- 48–66 Hz maximum
- 320 VA maximum

The automatic liquid sampler requires a proper earth ground. To protect users, the metal instrument panels and cabinet are grounded through the three-conductor power line cord in accordance with International Electrotechnical Commission (IEC) requirements.

The power cord must be plugged into a receptacle connected to a suitable earth ground. The receptacle ground should be verified.

WARNING

Any interruption of the grounding conductor or disconnection of the power cord could cause a shock that results in personal injury.

The controller is the power source for the injector and tray. The controller has an autoranging power supply.

Setting Up Installing the Injectors

Connecting the controller's power cord

1. Check the on/off button on the front of the controller. It must be off or flush with the front panel before you plug in the power cord. The symbol "⓪" indicates standby. The symbol "|" indicates on.
2. Plug the female end of the power cord into the AC power receptacle on the back of the controller.
3. Plug the male end of the power cord into the AC power receptacle of your facility.

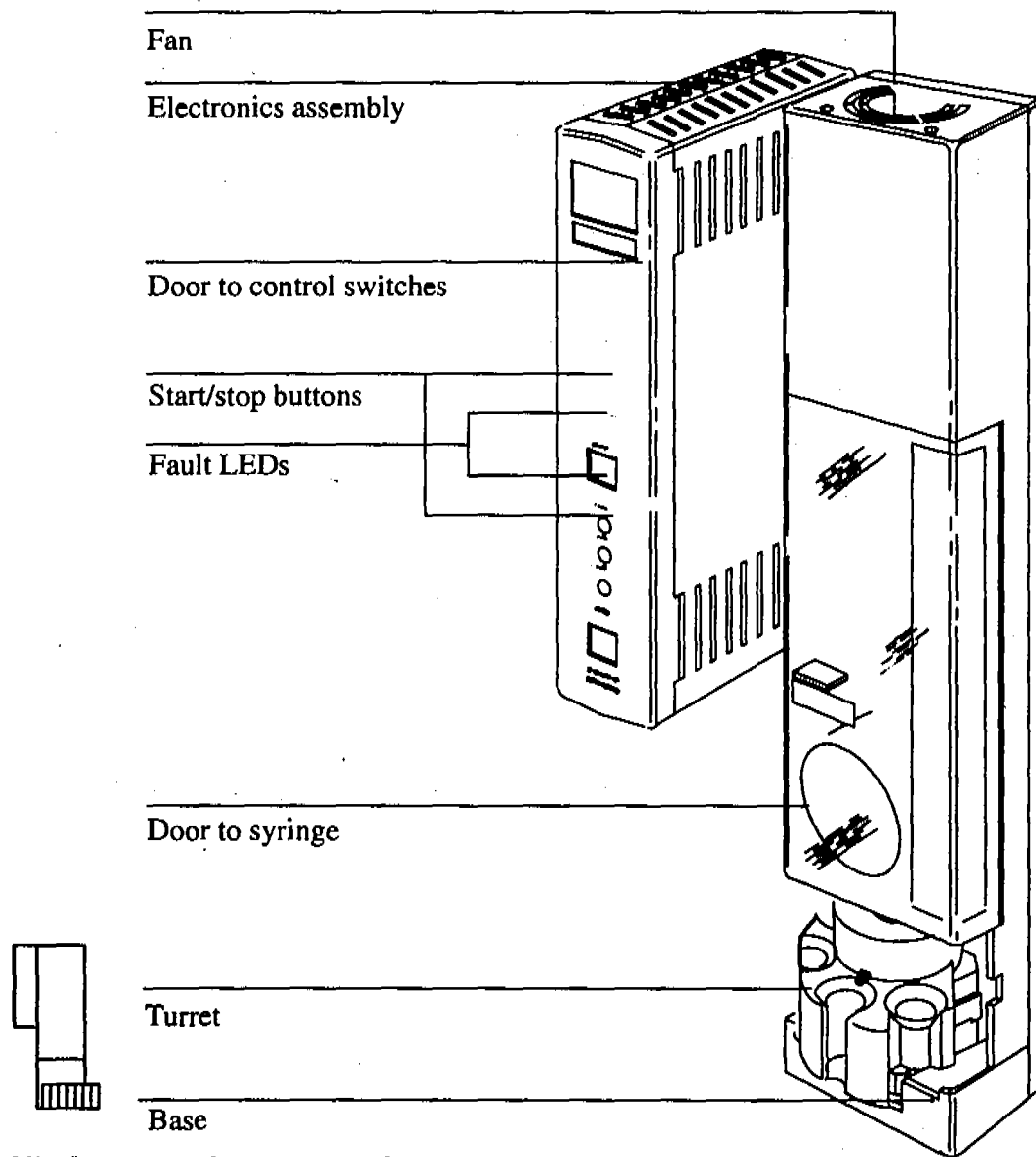
Installing the injectors

The injector contains the syringe, a syringe carriage, a six-position standard turret, a cooling fan that you can turn off, parameter switches, and a last sample vial switch.

The turret rotates sample vials and solvent and waste bottles into position under the syringe. Without a tray, you load the sample vials manually into the turret (maximum of three samples). With a tray, the robotic arm loads the vials (up to 100 samples). You load the solvent and wash bottles manually (the quantities of each depend on your controlling device). For more information on the fan, see "Using the injector fan" on page 39.

Setting Up Installing the Injectors

Figure 1-3



HP G1513A Injector module.

Behind the door to the control switches

There are control switches behind the door on the injector's front panel.

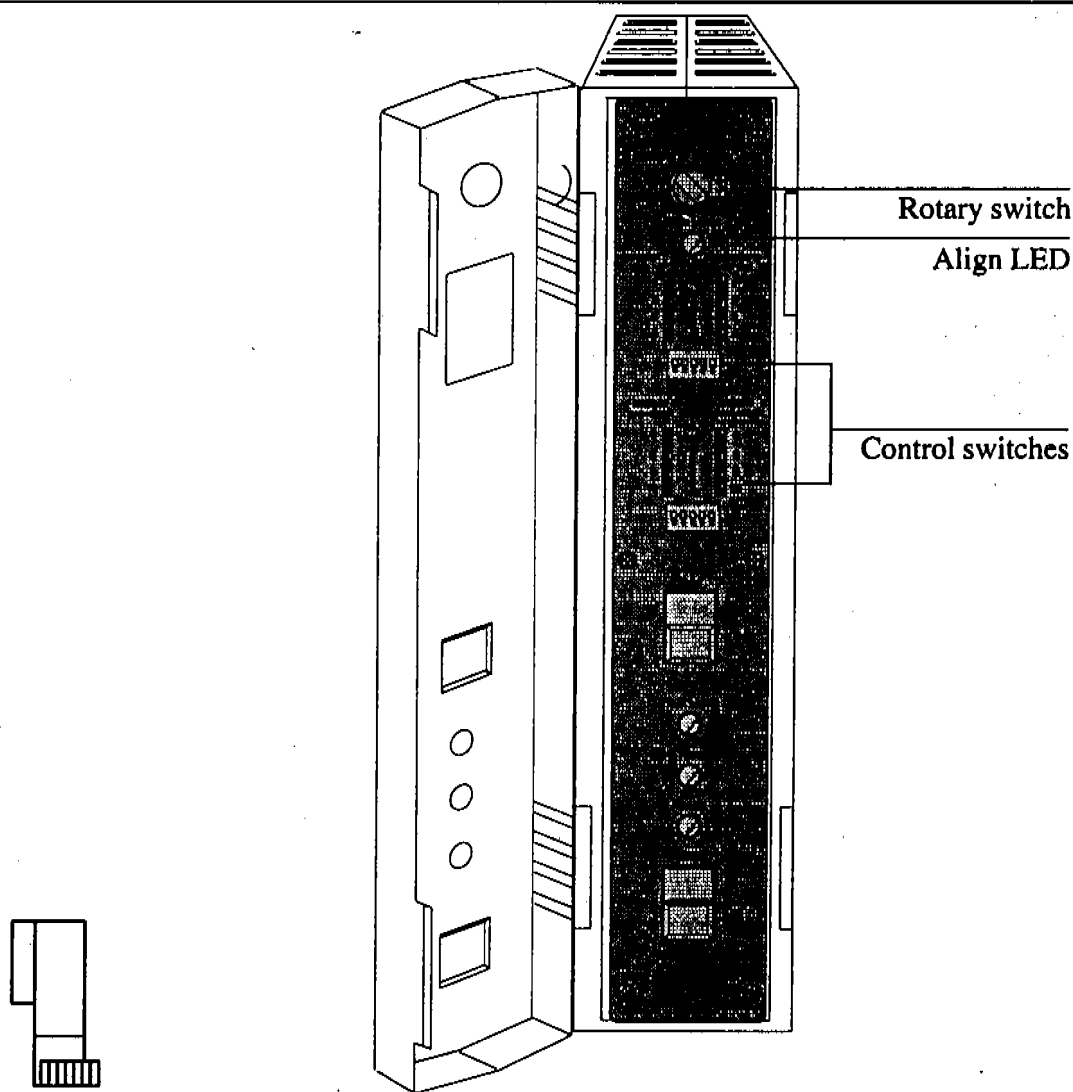
- You use the rotary switch to set the number of sample vials when no tray is installed and the remaining ten switches to set six injector parameters when the system cannot be controlled by the HP 6890 Series GC, a computer, or an integrator. For example, you can set the

Setting Up Installing the Injectors

number of injections per sample vial or the number of syringe washes. For more information, see "Setting the run parameters" on page 98.

- The injection switch can be used to set fast or slow injection with or without an integrator or computer controlling the system having any affect on that setting. However, it will be overridden by settings from the HP 6890 Series GC and the HP ChemStation.
- The Align LED is a diagnostic LED to warn users to perform the alignment procedure. See chapter 5, *Preventive Maintenance and Troubleshooting*.

Figure 1-4



The parameter control switches.

Setting Up Installing the Injectors

Before you start

To install an injector, you must first:

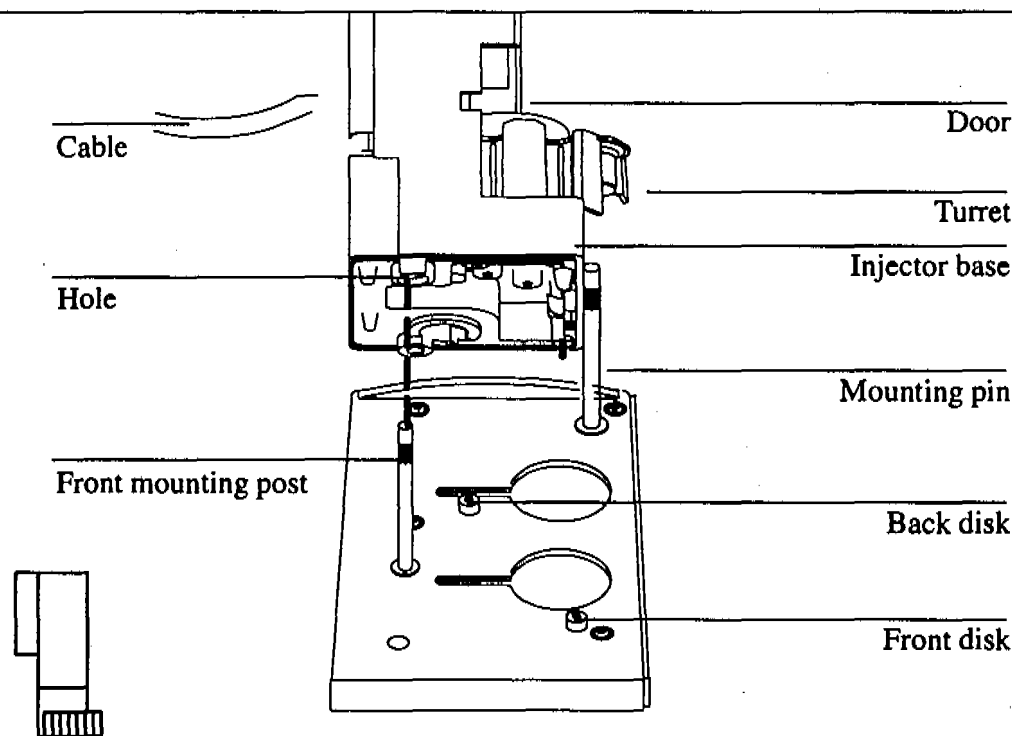
- Install the tray's mounting bracket before mounting the injectors (if your automatic liquid sampler includes a tray). The instructions and hardware are packaged together with the tray.
- Open the front panel, and remove the foam packing materials.
- Install the injector mounting posts in the front and back locations of the injection bracket on the GC.

Mounting the injectors

The first part of each step describes how to mount the injector over the front inlet (injection port).

1. Hold the injector with both hands so the cable points toward you. Line up the hole in the base of the injector that is nearest the cable with the mounting post on the bracket. Lower the injector about an inch onto the post.

Figure 1-5

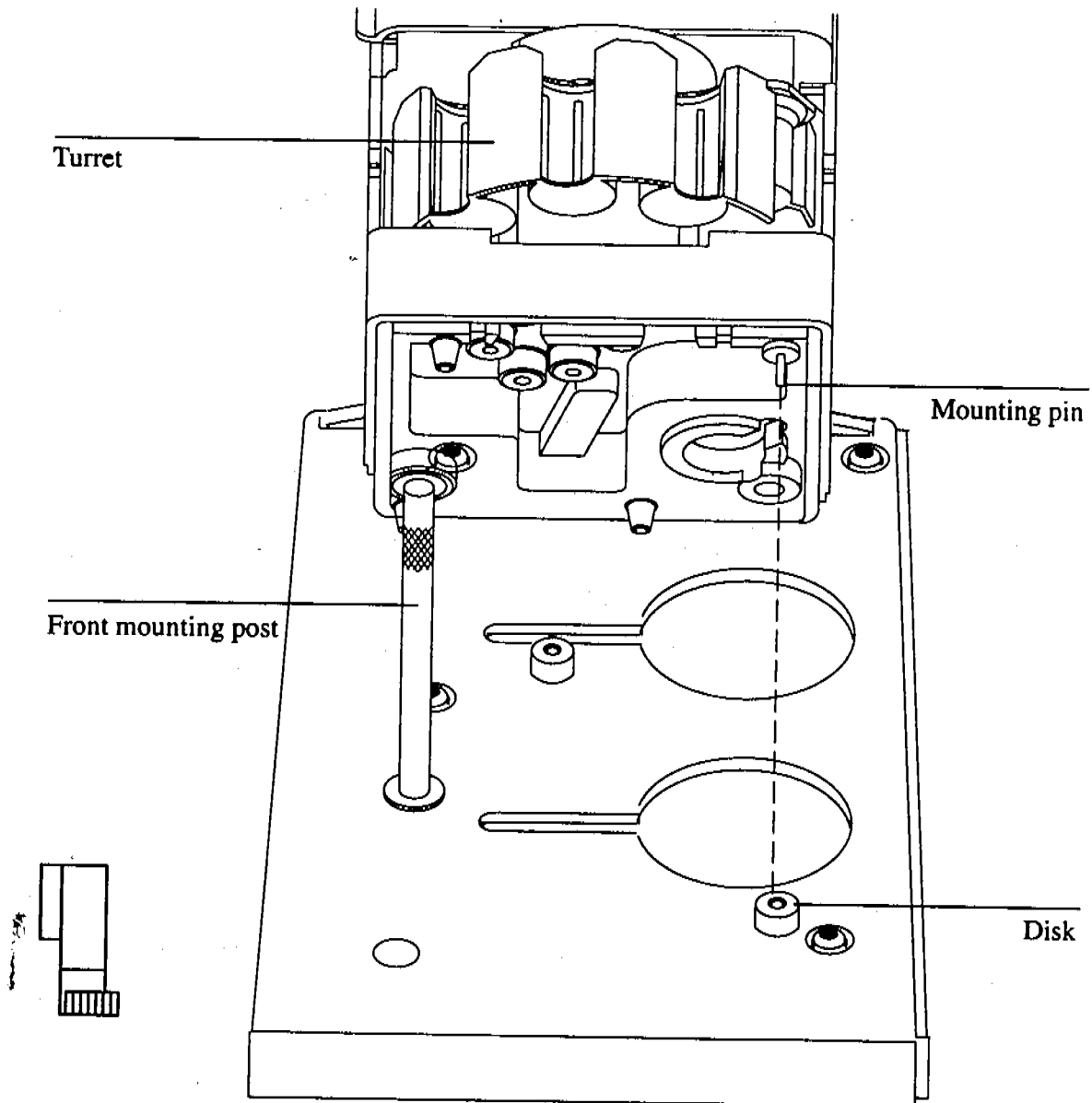


Lining up the injector with the mounting post.

Setting Up
Installing the Injectors

2. Turn the injector so that the turret is facing toward you. Lower the injector so that the pin in the base enters the hole in the disk on the mounting bracket.

Figure 1-6



Lowering the injector. The injector in this diagram is angled back to illustrate the position of the mounting pin.

Checking your work

- Be sure the mounting pin is seated in the hole of the disk. The injector's feet should touch the mounting bracket. Be sure the gas lines are not routed under the feet or the mounting pin.
- Turn the turret so you can see the inlet of the GC, and open the door to the syringe. Be sure the inlet is flush with the surface of the hole in the injector's base. This check does not apply to on-column inlets.
- If your system does not include a tray, read "Placing sample vials in the standard injector turret" in chapter 2.

For information on installing syringes, see "Selecting and installing syringes" in chapter 2. See this section for instructions on how to align the syringe to the inlet. Alignment is critical when injecting onto a 320- μm or 250- μm column. Chapter 6, "Special Topics," contains more information on use of 320- μm and 250- μm columns.

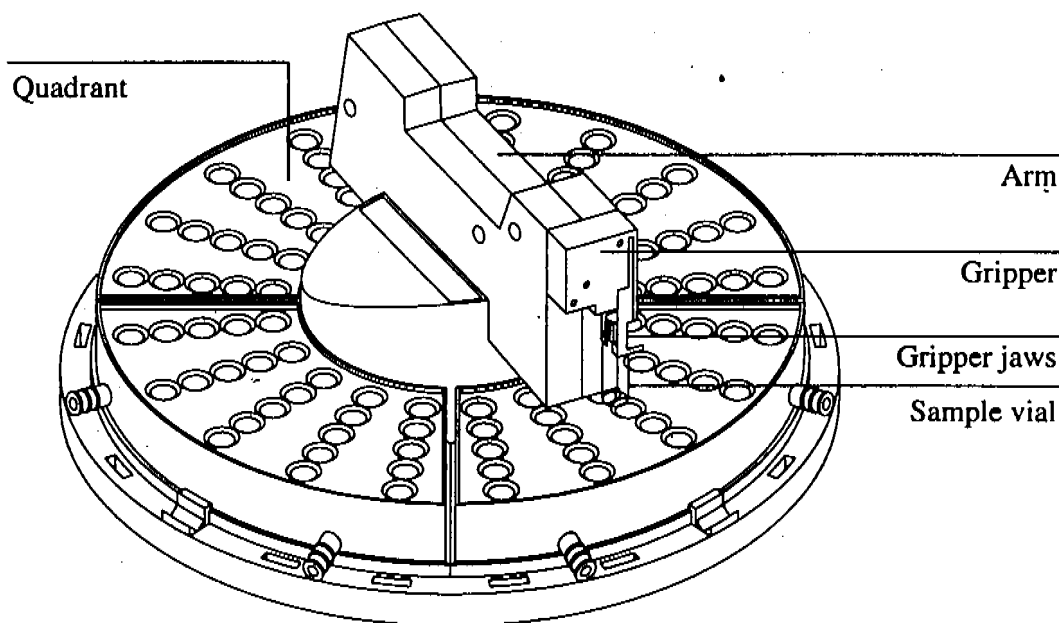
Installing the tray

The tray delivers samples to one or two injectors depending on the configuration of the gas chromatography system. The arm and gripper mechanism in the center of the tray loads sample vials into the injector turret and then returns them to the tray after each injection. It can also transport sample vials to and from the HP G1296A/HP G1926A bar code reader.

Each of the four removable tray quadrants holds 25 vials. Each is hollow and has fittings so that you can circulate a temperature-controlled fluid through it.

Setting Up
Installing the tray

Figure 1-7



HP 18596B tray module.

Before you start

Caution

Do not move the tray's arm back and forth. Do not move the tray's gripper up and down. These movements could damage the tray arm. If you need to move the arm, turn it in a clockwise or counterclockwise direction.

To install a tray, you must first:

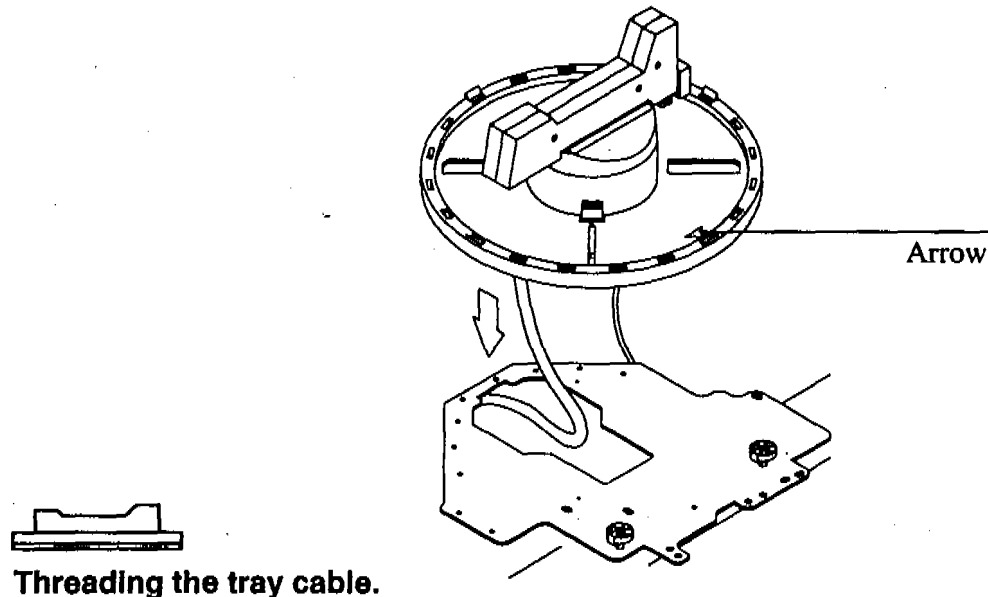
- Install the tray mounting bracket. The mounting bracket for the tray attaches to the injector mounting bracket and the GC. The instructions and hardware are packaged together with the tray.

Setting Up
Installing the tray

Mounting the tray

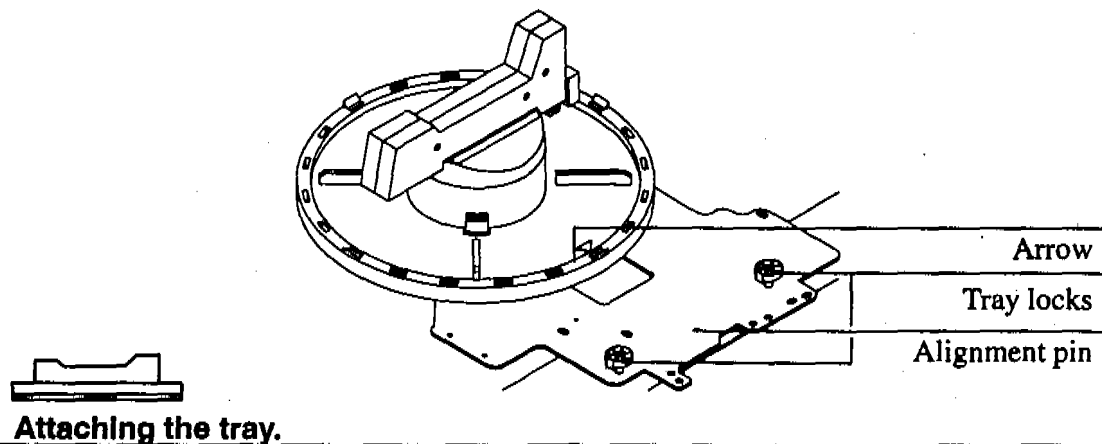
1. Thread the cable through the hole in the mounting bracket. Lower the tray onto the bracket. Position the tray so that it sits flat on the bracket with the raised arrow pointing toward the right (the front injector). Move the injector cable out of the way.

Figure 1-8



2. Line up the arrow on the tray base with the alignment pin on the mounting bracket. Slide the tray to the right until the edge is under the two tray locks.

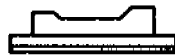
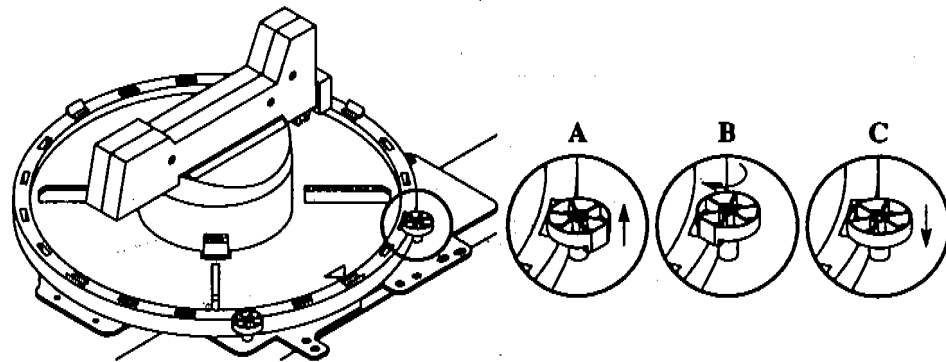
Figure 1-9



Setting Up
Installing the tray

3. Lock the tray into place by pulling each lock up (A), turning each lock (B), and inserting each tab into a slot in the tray (C).

Figure 1-10

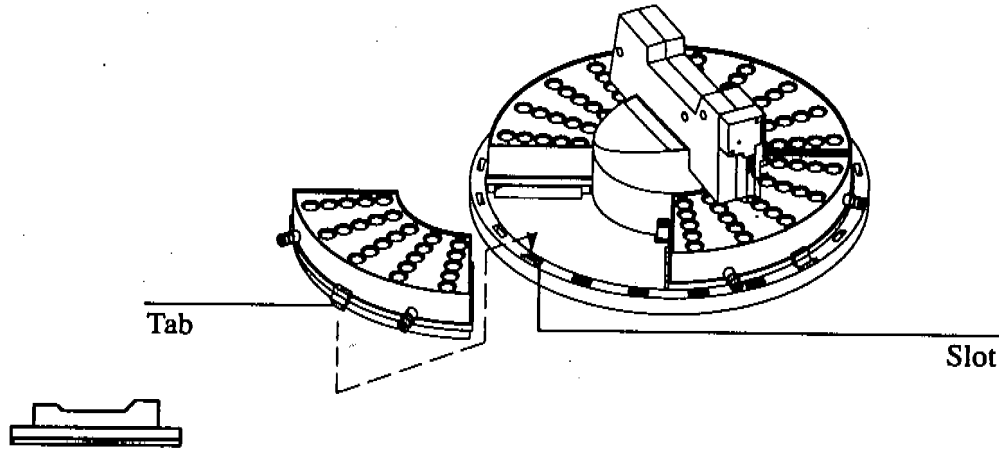


Locking the tray.

**Setting Up
Installing the tray**

4. Snap the tab of each tray quadrant into a slot on the base of the tray.
The numbers on the quadrants should match the numbers on the base.

Figure 1-11



Inserting the tray quadrants.

Checking your work

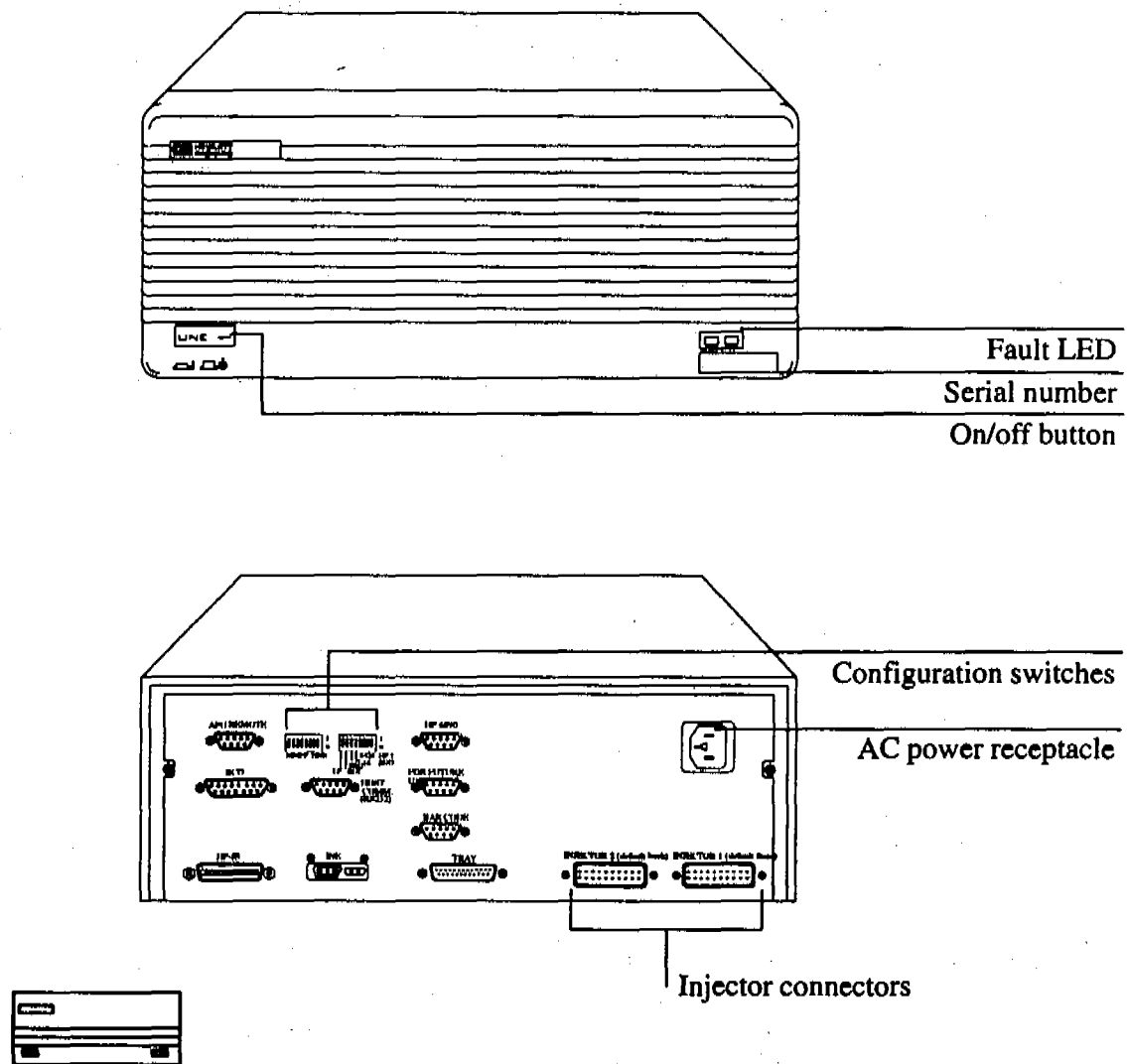
- Be sure the tray base is all the way to the right and locked into place.
- Be sure the quadrants are seated on the tray base.

Installing the controller

The HP G1512A controller provides power and the communication interface for HP 18593A/B or HP G1513A injectors and for the HP 18596A or HP 18596B/C tray.

There are 11 connectors and two sets of switches for defining the communication type.

Figure 1-12



HP G1512A controller and communications module.

Setting Up Installing the controller

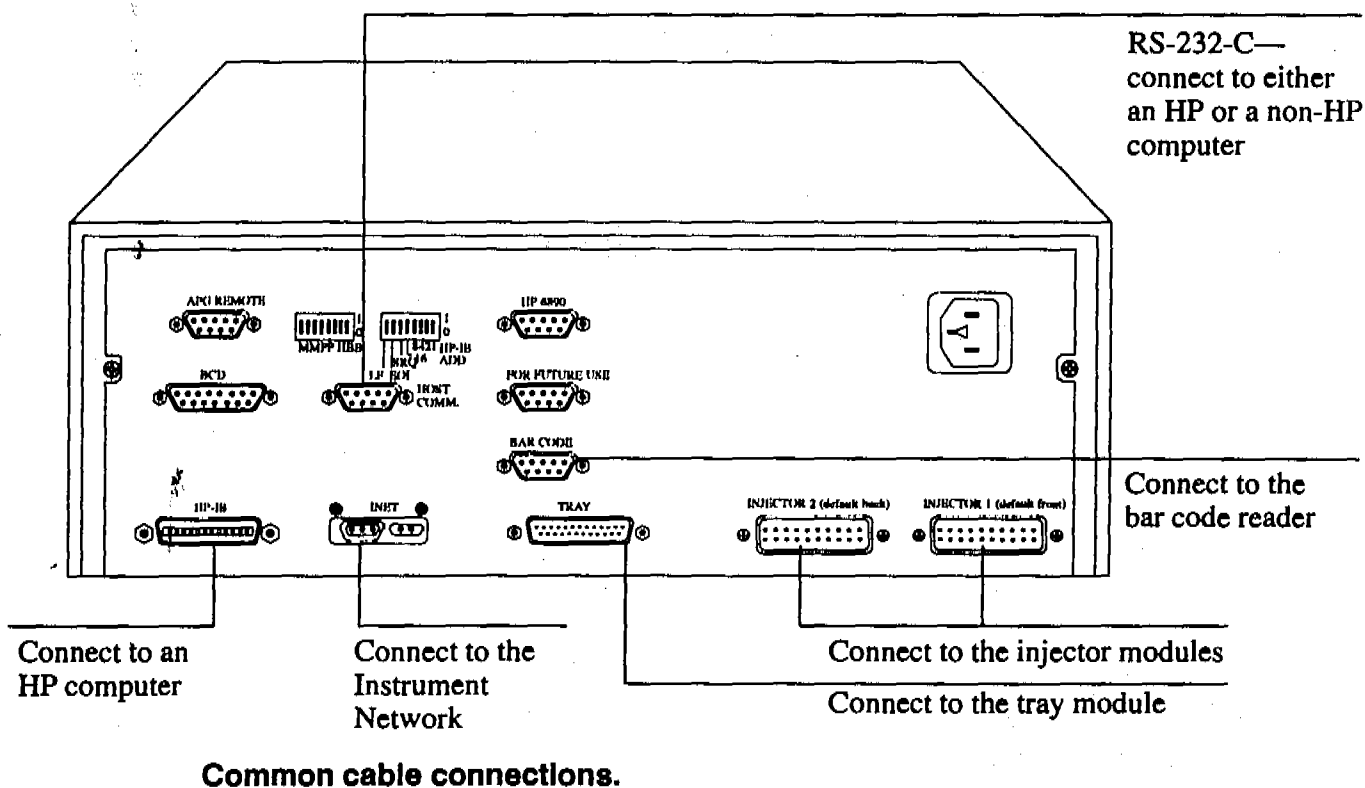
This section covers connecting the injector and tray cables to the controller, connecting the controller to the GC, connecting the controller to the data handling device, and plugging in the controller.

Find the power cord in the controller box. Check the shape of the plug and the source voltage listed on the packing contents sheet. Verify that the power cord is appropriate for the power source at your facility.

Connecting cables to other instruments

The automatic liquid sampler communicates to other instruments via cables that connect to the various connectors on the controller. Figure 1-13 shows the connectors on the back of the controller and what they are used for.

Figure 1-13



Setting Up Installing the controller

Setting the configuration switches

There are 16 switches on the back of the controller. Figure 1-14 shows the switches and settings for five common types of communications.

When you are using HP ChemStation software, you need to set the HP-IB address switches to a unique address. The address for the first automatic liquid sampler is usually set to "8." For a complete list of address switches, see the next section.

Configuration switch definitions

The following tables describe the configuration switches and their settings.

Left-hand Switch Settings

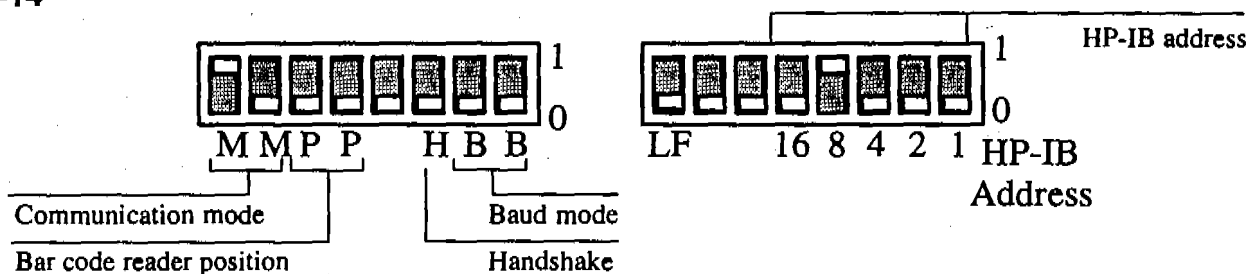
| Switch | Description | Possible Values |
|--------|------------------------------|--|
| MM | Communication mode type | 00—INET, standalone 10—3365 ChemStation, MS ChemStation (Rev 3.65 and lower) 11—HP ChemStation, MS ChemStation (Rev 3.71 and higher) 01—Asynchronous standalone |
| PP | The bar code reader position | 00—Front 01—Back 10—Right 11—Left |
| H | RS-232-C host handshake | 0—XON/XOFF 1—RTS/CTS |
| BB | RS-232-C host baud rate | 00—2400 01—9600 10—19.2 K 11—38.4 K |

Right-hand Switch Settings

| Switch | Description | Possible Values |
|------------|--------------------------------|---|
| LF | Controller termination message | 0—Line feed only 1—Carriage return and line feed |
| 16,8,4,2,1 | HP-IB address of controller | 00000=0, 00001=1, 00010=2, 00011=3, etc (see figure 1-14) |

Setting Up
Installing the controller

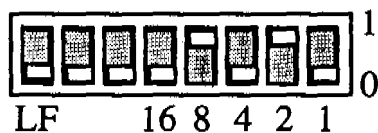
Figure 1-14



Setting the address switches.

Figure 1-15

HP ChemStation
HP 6890 Series GC
MSD ChemStation
(rev 3.70 and above)



Address = 10

HP 3365 ChemStation
MSD ChemStation
(rev 3.65 and below)
HP-UX ChemStation

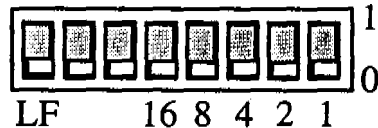


Address = 8

HP Integrator w/INET
Synchronous standalone



Asynchronous standalone



HP 35900 loop



RS-232-C, described in
serial interface manual
(HP p/n 18594-90300)



Examples of switch settings.

Setting Up
Installing the controller

Possible HP-IB Address Switch Settings

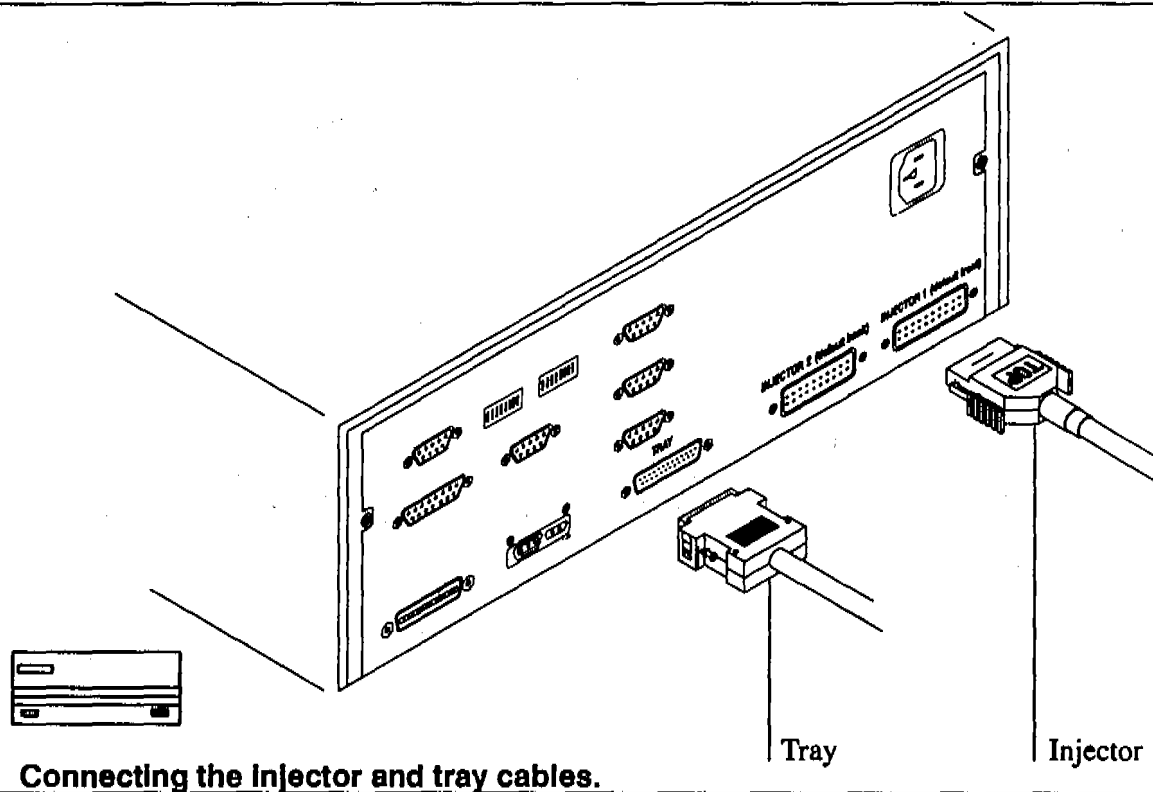
| <u>HP-IB Address</u> | <u>Switches</u> |
|--------------------------|-----------------|
| 0 | 00000 |
| 1 | 00001 |
| 2 | 00010 |
| 3 | 00011 |
| 4 | 00100 |
| 5 | 00101 |
| 6 | 00110 |
| 7 | 00111 |
| 8 | 01000 |
| 9 | 01001 |
| 10 | 01010 |
| 11 | 01011 |
| 12 | 01100 |
| 13 | 01101 |
| 14 | 01110 |
| 15 | 01111 |
| 16 | 10000 |
| 17 | 10001 |
| 18 | 10010 |
| 19 | 10011 |
| 20 | 10100 |
| 21 | 10101 |
| 22 | 10110 |
| 23 | 10111 |
| 24 | 11000 |
| 25 | 11001 |
| 26 | 11010 |
| 27 | 11011 |
| 28 | 11100 |
| 29 | 11101 |

Setting Up Installing the controller

Connecting the injector cables

With the power off, plug each injector cable into the connector on the back panel of the controller associated with the position of the injector, front or back. Be sure the spring clamps on either side of the plug snap onto the connector. Note the TOP label on the connector for orientation purposes.

Figure 1-16



Connecting the injector and tray cables.

Connecting the tray cable

Plug the tray cable into the tray connector on the back panel of the controller. Secure the plug to the connector with a small flathead screwdriver.

Setting Up Installing the controller

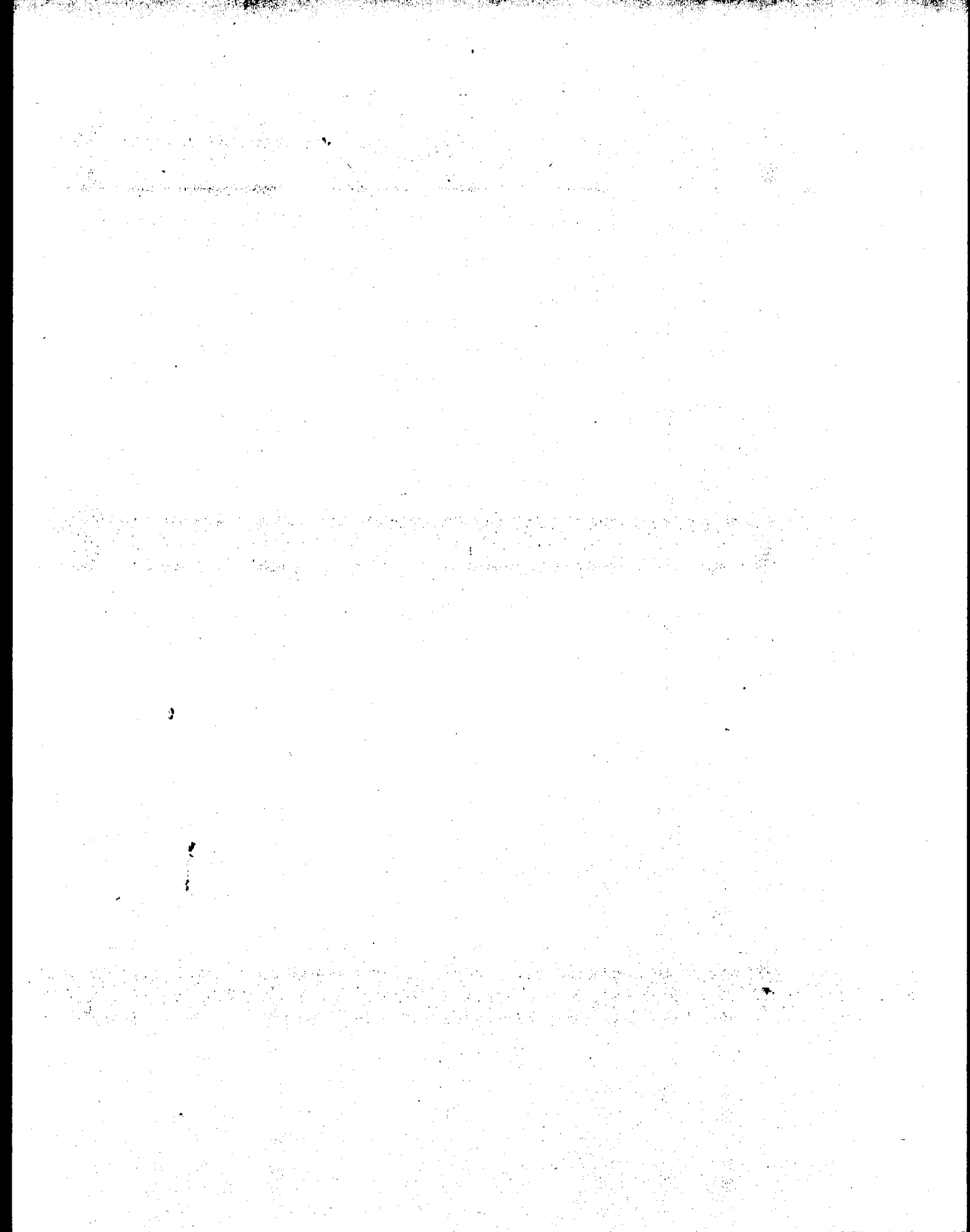
Connecting the controller's power cord

1. Check the on/off button on the front of the controller. It must be off or flush with the front panel before you plug in the power cord.
2. Plug the female end of the power cord into the AC power receptacle on the back of the controller.
3. Plug the male end of the power cord into the AC power receptacle of your facility.

Checking your work

- Be sure the injector and tray plugs are fastened securely.
- Be sure all the communication cables are installed and fastened securely.
- Be sure any external control or data handling devices are configured correctly by referring to the appropriate manual.
- Turn the controller on. Listen for a single beep. This means the system initialized correctly.
 1. On the injector, the red, yellow, and green lights flash on together. The red and yellow lights go off. The green light stays on. If the red light stays on, be sure the injector is mounted correctly and the door to the syringe chamber is closed.
 2. On the controller, the yellow and green lights flash on together. The green light stays on while the yellow light goes off.

To test the operation of the automatic liquid sampler, turn to chapter 2, "Preparing for Operation."



Preparing for
Operation

Preparing for Operation

This chapter contains detailed information for preparing the automatic liquid sampler for operation, including:

- Preparing sample vials
- Using the injector fan
- Preparing the solvent and waste bottles
- Selecting and installing syringes
- Maintaining the inlet
- Adapting for cool on-column injection

To optimize your sampler operation, it is important to have thorough preparation of your samples, sample vials, syringes, and inlets. Regular maintenance also keeps your equipment and analysis running smoothly. For more information, see chapter 5, "Preventive Maintenance and Troubleshooting."

Preparing sample vials

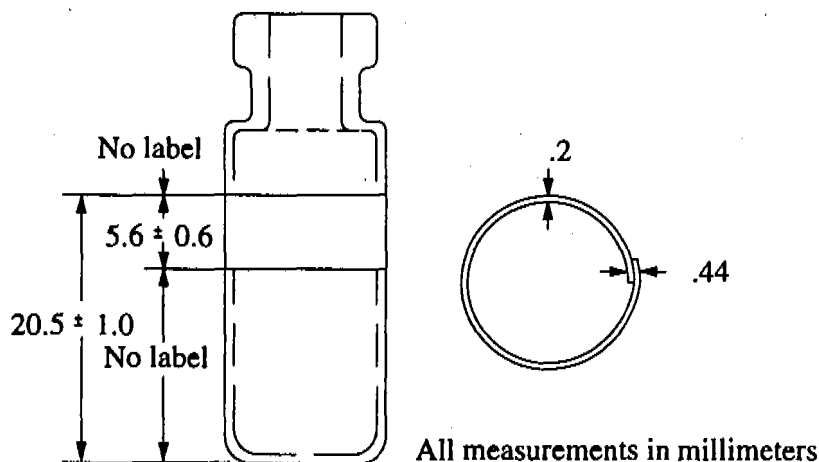
This section explains how to select, label, fill, cap, and place the sample vials.

Selecting and labeling sample vials

The injector and the tray use glass sample vials and crimp caps or Target® DP™ screw-cap vials that meet a set of specifications. These are available with a write-on spot for easy labeling. If you choose to make your own labels, read the following specifications for location and thickness of labels.

The location and thickness of a vial label can affect the delivery of the bottles to and from the injector. Hewlett-Packard recommends the position and maximum label thickness shown in the following diagram.

Figure 2-1



Label specifications.

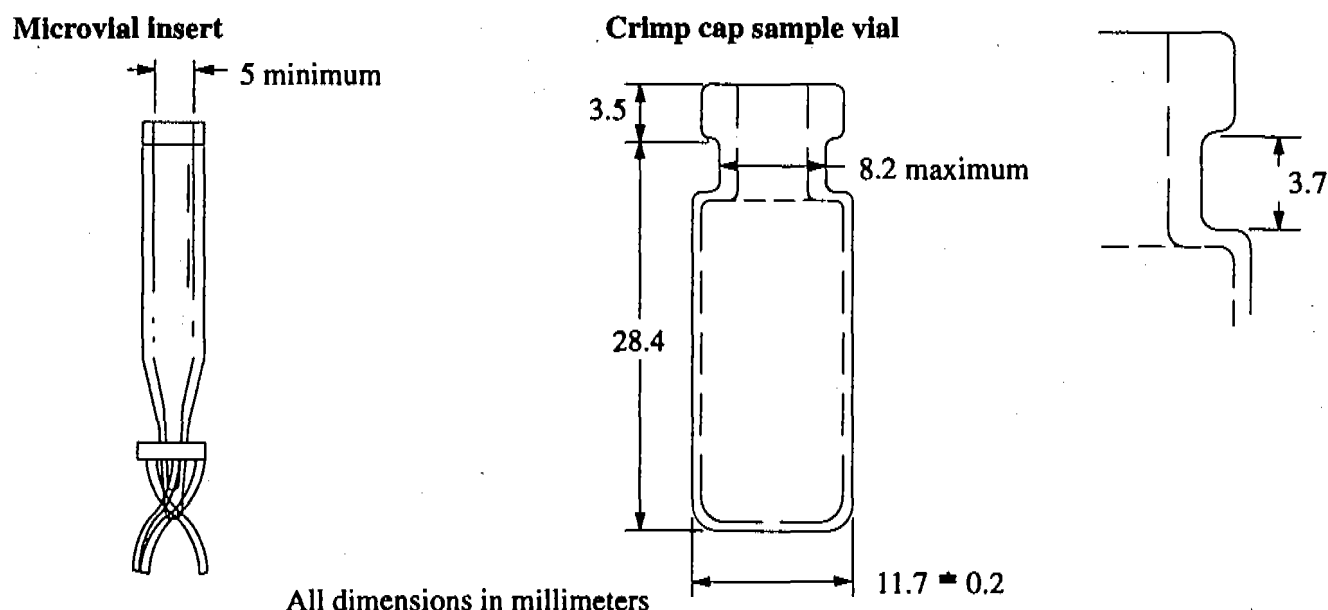
Caution

The correct sample vial dimensions are critical for proper operation. Vials that do not meet specifications may cause sampler errors. Service calls and repairs found to be due to vials and microvials that do not meet these specifications are not covered under warranty or the service contract.

Specifications

Figure 2-2 illustrates most of the critical dimensions for the sample vials and microvial inserts. These dimensions do not make up a complete set of specifications. Some of the dimensions are too difficult to measure without special instruments.

Figure 2-2



Dimensions for sample vials and microvial inserts.

Sample caps and septum

Although septa come in different colors, the characteristics are measured by inertness and type of composition. There are two basic types of septa used with both crimp caps and screw-on caps. Each has different resealing characteristics and a different resistance to interaction with solvents.

- A general-purpose rubber formulation made from natural rubber is coated with Teflon on the sample side. It has a pH range of 4.0–7.5. It is least resistant to solvents after puncture, however, it is more easily cored. This coring may place septum pieces in the solvent that affect your chromatograms.

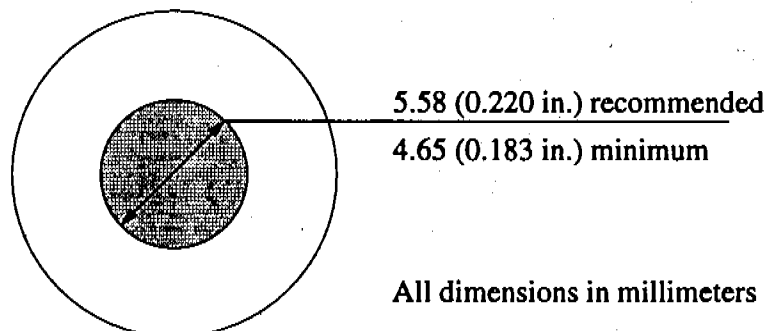
Preparing for Operation
Preparing sample vials

- A high-quality, low-extractable silicone rubber, coated with Teflon on one or both sides, is somewhat resistant to solvents after puncture.

Refer to your *Analytical Columns and Supplies Catalog* for more information.

Figure 2-3 illustrates the recommended and minimum diameter for vial cap apertures.

Figure 2-3

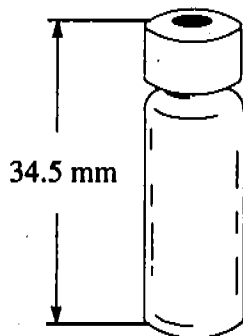


Specifications of vial cap aperture.

Use amber glass vials for light-sensitive samples.

The last specification to consider is the total height of the capped vial. Figure 2-4 illustrates the recommended maximum height of a crimp capped or screw capped vial.

Figure 2-4



Maximum height of a vial with cap.

Preparing for Operation

Preparing sample vials

Filling sample vials

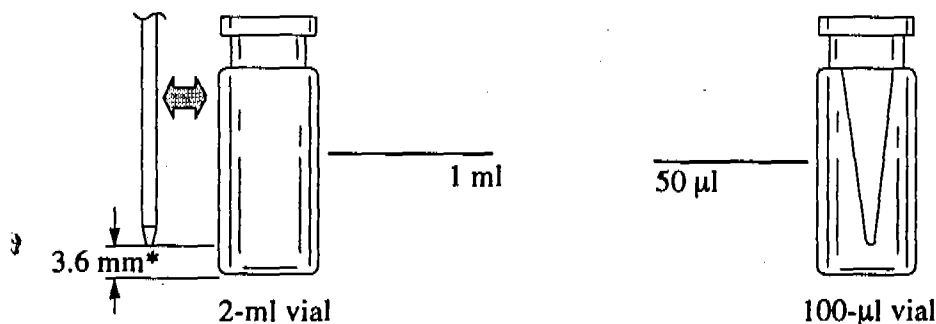
Follow these recommendations to obtain reliable, high performance with the sampler and prevent contamination or injection volume problems.

Recommended volumes for a maximum of five injections per vial are:

- 1 ml for the 2-ml vial
- 50 μ l for the 100- μ l vial

For large volumes and multiple injections, you will have to decide how to divide the sample among several vials to obtain reliable results. You have to be aware when sample volume is low. For example, if the vials are less than half full, contaminants from the previous sample injection or solvent washes may affect the sample. Due to the various parameter settings available, the many suppliers of consumables, and the choices of vials, microvials, vial caps, and septa, some method development will need to be done to optimize your analysis.

Figure 2-5



*Needle position based on standard sampling depth. See chapter 3 for more information on setting parameters for sampling depth.

Recommended volumes for sample vials.

Caution

If the vials are more than half full, a vacuum may interfere with the syringe delivering a precise volume.

Do not inject air into the vial to compensate for the vacuum. Injecting air into the vial often damages the cap septum so that it is no longer airtight.

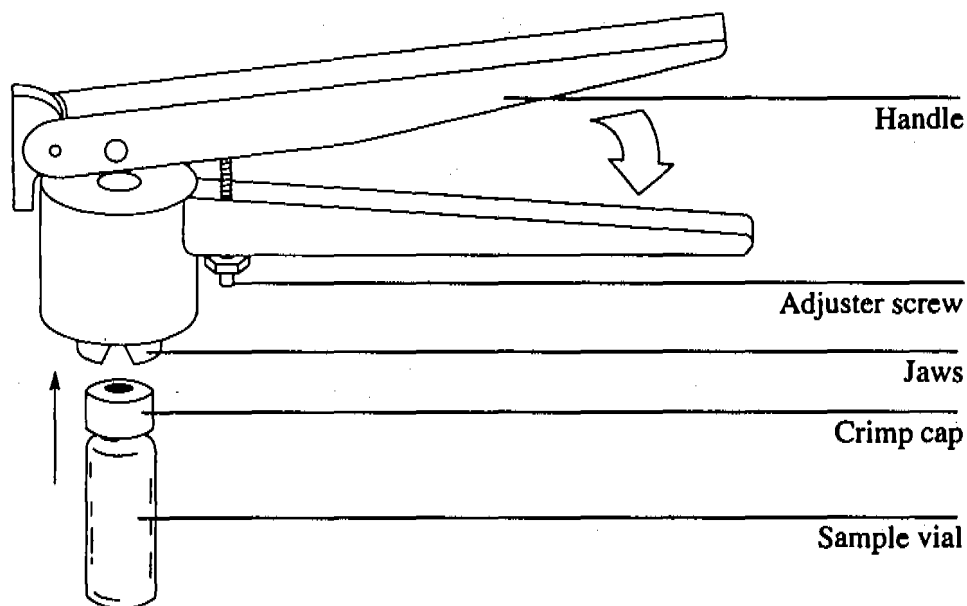
Preparing for Operation
Preparing sample vials

Crimp capping sample vials

Use a crimper to put on the airtight crimp caps.

1. Clean off the inside surfaces of the crimper jaws.
2. Place the crimp cap over the top of the vial.
3. Lift the vial into the crimper, and squeeze the handle until the bottom grip reaches the adjuster screw.

Figure 2-6



Crimping caps.

Caution

Vials that do not have properly crimped caps may cause sampler errors.

When a tray is not installed, you may be able to use sample vials with no caps, snap-on caps, or screw-on caps depending on your application. If a tray is installed, sample vials must have crimp caps or Target® DP™ screw-cap vials.

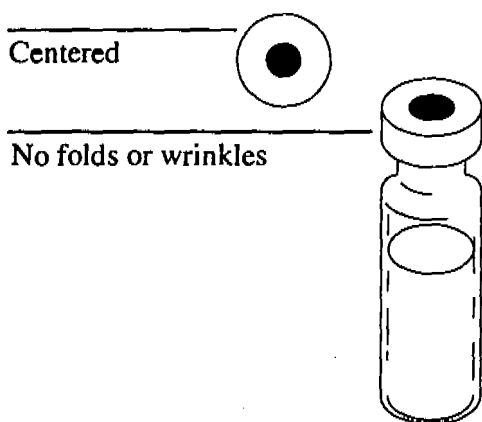
Preparing for Operation
Preparing sample vials

Check that your vial is crimped as follows:

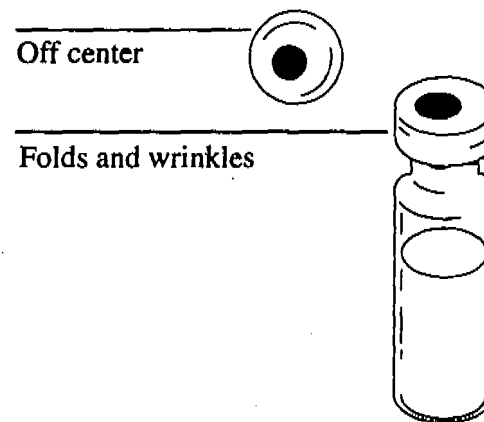
1. Cap has no metal folds or wrinkles on the part of the cap that wraps under the neck of the vial.
 - If there are folds or wrinkles, flatten any wrinkles by turning the vial about 10° and crimping it again. Adjust the crimper for a looser crimp by turning the adjusting screw clockwise.
2. Cap is too tight to turn by hand.
 - If the cap is loose, adjust the crimper for a tighter crimp by turning the adjusting screw counterclockwise. Crimp the cap again.
3. Cap has a flat septum centered over the top of the vial.
 - If the septum is not flat, remove the cap, turn the adjusting screw clockwise, and try again.
 - If the cap is not centered, remove the cap, and make sure the new cap is flat on the top of the vial before you squeeze the crimper.

Figure 2-7

Acceptable



Unacceptable



Acceptable and unacceptable caps.

Preparing for Operation

Preparing sample vials

There are three reasons for crimping the cap properly:

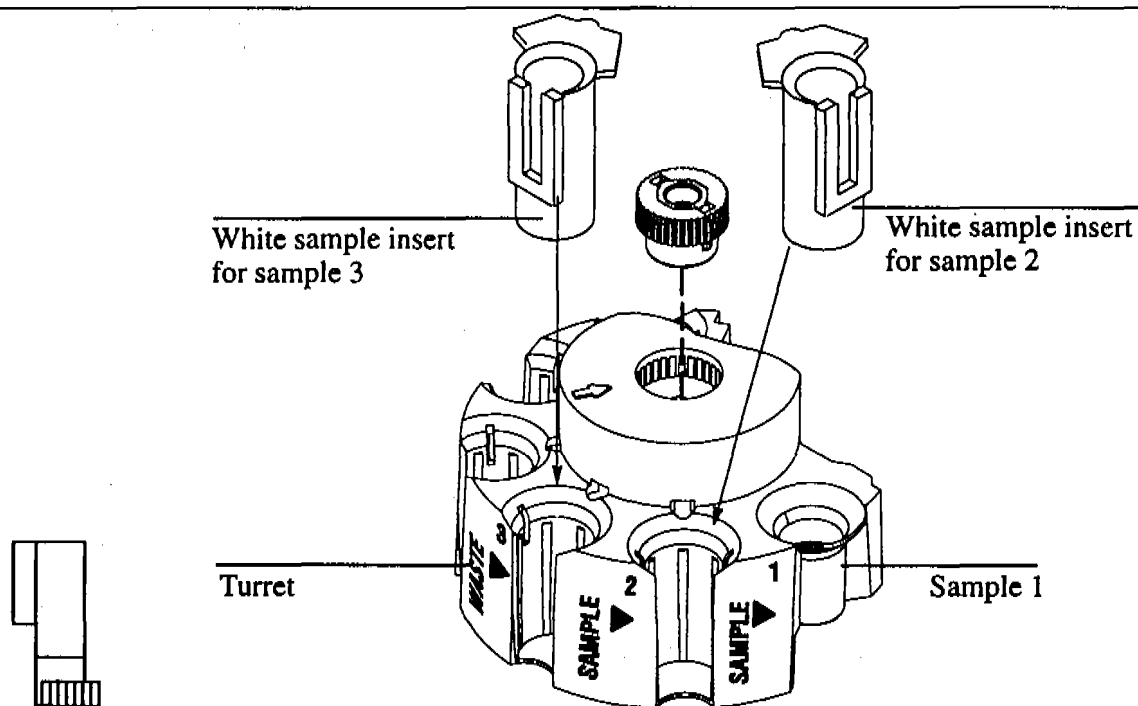
- The syringe tends to core a curved vial septum and drop small pieces of the material into the sample.
- The syringe needle could hit the metal part of the uncentered cap.
- The tray gripper may drop a vial if the cap has folds or wrinkles in it.

Placing sample vials in the standard injector turret

If you do not use the tray, you can place one, two, or three vials in the injector turret. For two or three sample vials, you must convert some of the bottle positions to sample vial positions with sample inserts.

1. Place the appropriate sample inserts in bottle positions 2 and 3 on the turret.
 - For one sample, no inserts are needed. Position one is molded into the turret.
2. Place the sample vials into the sample inserts.

Figure 2-8



Placing sample Inserts for three sample vials.

Placing sample vials in a tray

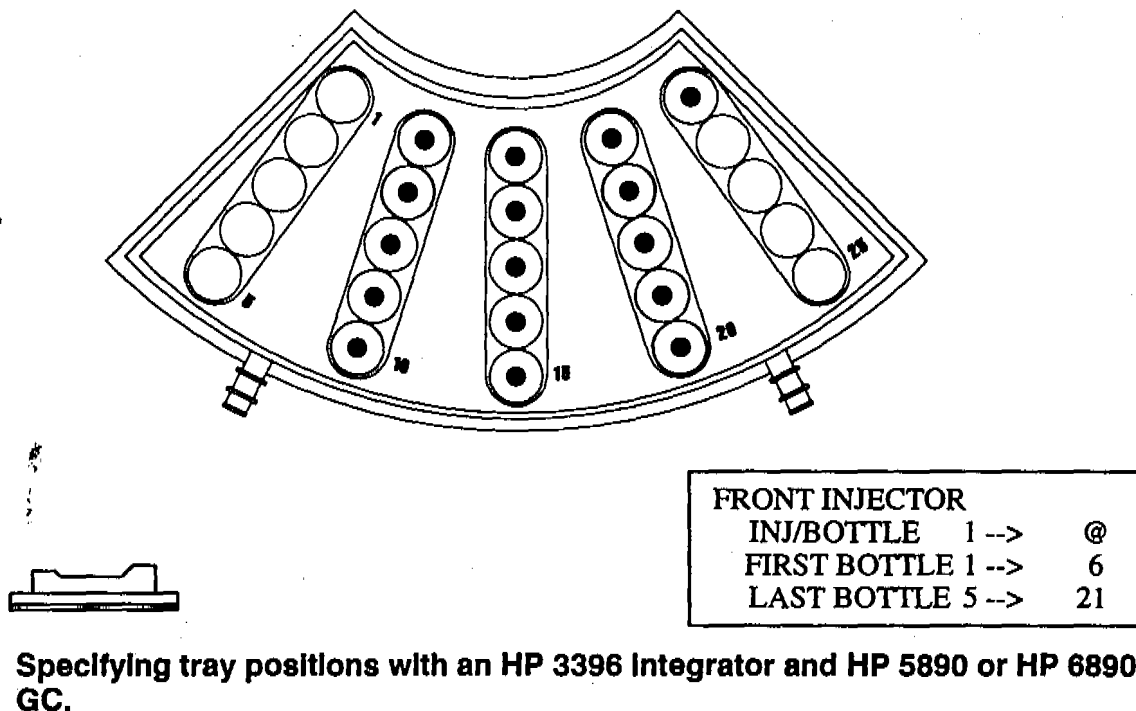
You can place up to 100 samples in the tray's four quadrants. The tray gripper picks up the vials and delivers them to the sample vial position in the turret.

When you are not using the external control instruments, place the first bottle in quadrant position 1. The tray continues to deliver vials until it delivers the last one in the series or until it encounters an empty position. For more information, see chapter 4, "Standalone Control."

When you are using another instrument such as an integrator or ChemStation to control the tray, the first and last bottle positions are defined when you set the sequence parameters. The tray begins picking up sample vials at the position that corresponds to the "first bottle" and stops after it replaces the vial that corresponds to the "last bottle."

For more information, see "Setting the run parameters" in chapter 3.

Figure 2-9



Using the injector fan

The purpose of the fan is to push cool air over the samples and solvent to keep them cooler. Also, some samples may boil out of the syringe if heat builds up in the injector.

For a particular application, you may want the area around the samples to be warm. Heat can help the delivery of viscous, high-boiling samples.

The default position of the fan is on. If your analysis requires the fan to be off, use the instructions "Turning the fan off" below.

Turning the fan off

To turn the fan off, follow these steps.

1. Turn the controller power switch off.
2. Put on a grounded wrist strap.

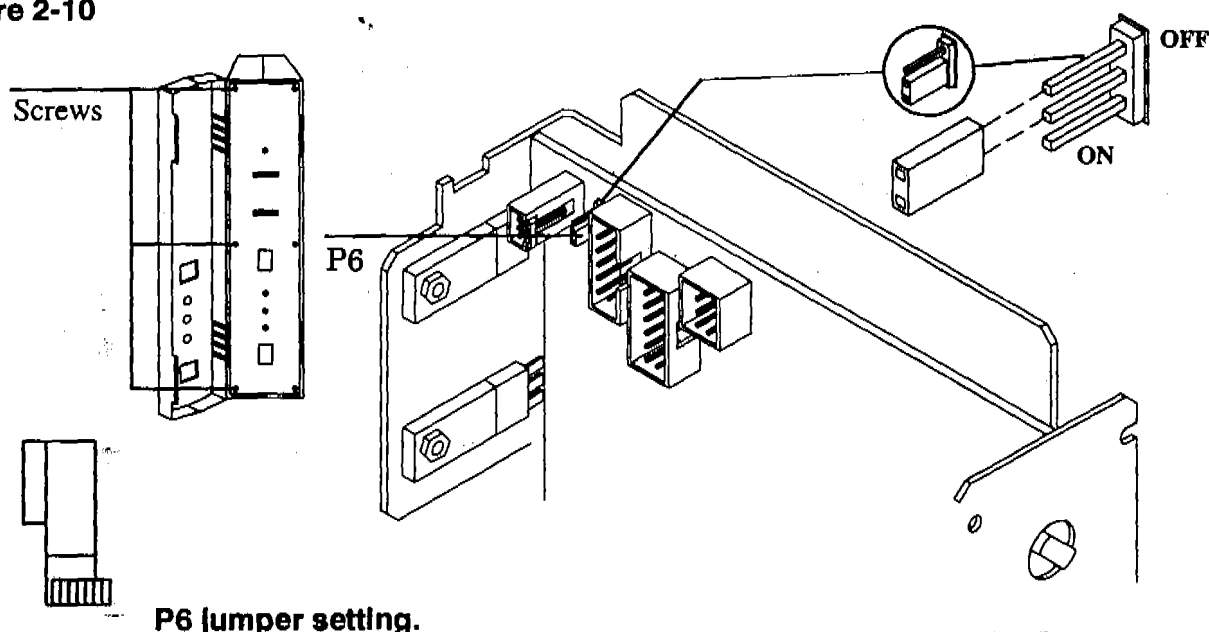
Caution

This procedure requires protection against electrostatic discharge. Use a static control wrist strap connected to a ground (HP part no. 9300-0969 for large wrists or HP part no. 9300-0970 for small wrists). If you do not use static protection, you may damage the electronics of the injector. Do not touch any of the electrical components, especially the microprocessor.

3. Open the door to the injector electronics assembly, and remove the three screws on the left edge of the panel with a No. 1 Point Pozidrive screwdriver.
4. Open the left-hand side of the assembly. Remove the blue ribbon cable (P3) so P6 is more visible. Locate the P6 jumper switch on the top left front corner of the printed circuit board.
5. Connect the P6 jumper so that it covers the top two prongs on the circuit board labeled **OFF**. See figure 2-10. Return the ribbon cable to its original position.

Preparing for Operation
Preparing the solvent and waste bottles

Figure 2-10



6. Close the left-hand side of the assembly, and replace the three screws on the left edge of the front panel.
7. Restore the power. The fan should now be off. If it is still running, reopen the injector, and check the position of the P6 jumper according to the instructions in step 5.

3 Preparing the solvent and waste bottles

The solvent bottles hold solvent for rinsing the syringe between injections. The injector dispenses the solvent washes and sample washes into waste bottles.

The first two parts of this section explain how to select, fill, and place the solvent and waste bottles. The last two parts of this section explain how to estimate the maximum number of vials you can run at one time and how the injector controls carryover.

For information on how to set the number of washes, see "Setting the run parameters" in chapter 3.

Preparing for Operation

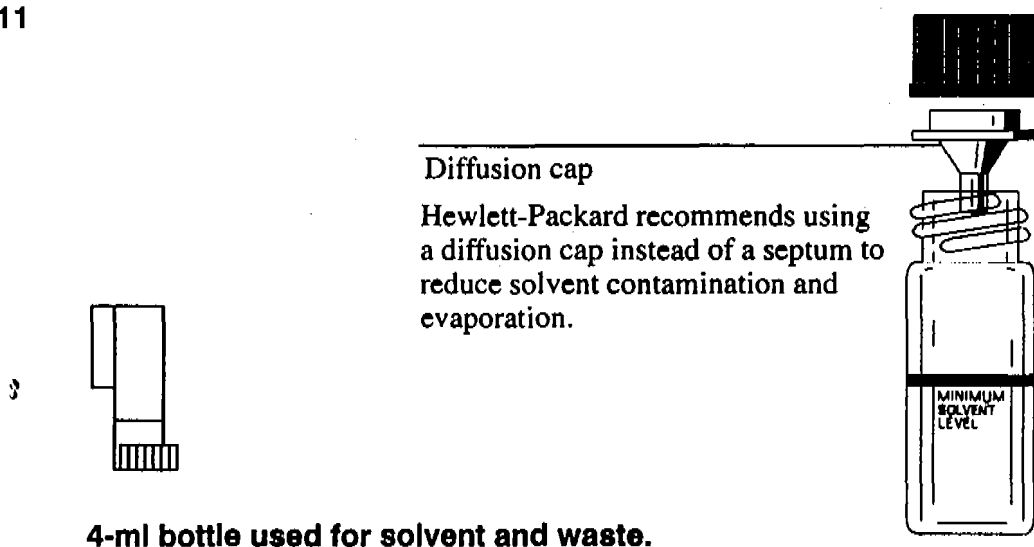
Preparing the solvent and waste bottles

Selecting the bottles

The injector uses 4-ml bottles with diffusion caps to hold the solvent and waste. You can use diffusion caps or septa on these bottles to reduce evaporation and diffusion of your solvents and waste. Hewlett-Packard recommends diffusion caps over septa for two reasons:

- The diffusion cap allows multiple entrances into a bottle without contaminating the liquid inside the bottle with small pieces of septum material.
- For many common solvents, the rate of diffusion out of the bottle is less with a diffusion cap than with a septum that has been punctured with a standard syringe needle.

Figure 2-11



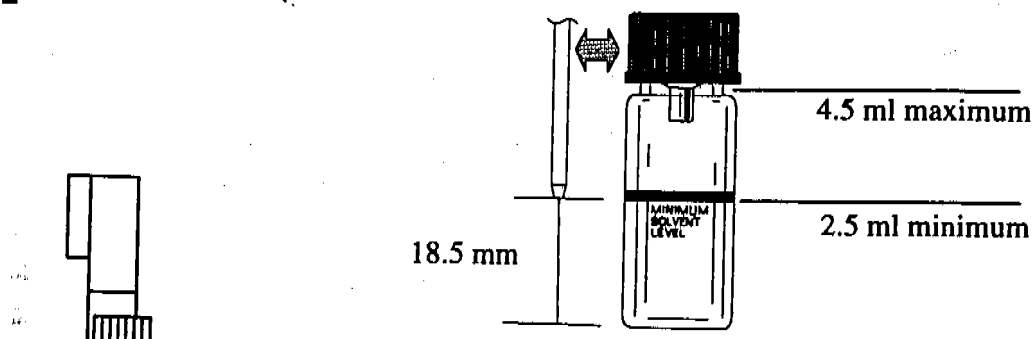
Filling and placing the bottles

Before each sequence or group of sequences, prepare your solvent and waste bottles by doing the following:

1. Rinse and fill each solvent bottle with 4 to 4.5 ml of fresh solvent. The liquid level should be near the shoulder of the bottle. If the solvent bottle is filled with 4.5 ml of solvent, the syringe can reach about 2 ml or about 250 washes for a 10- μ l syringe.

Preparing for Operation
Preparing the solvent and waste bottles

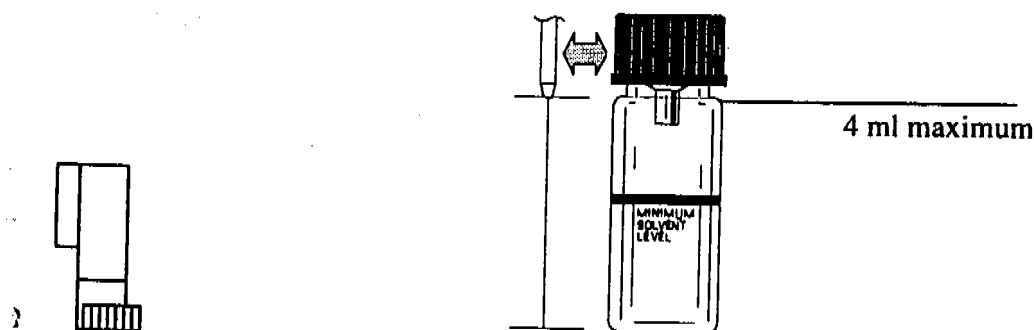
Figure 2-12



Shows position of the syringe tip when withdrawing drawing solvent.

2. Empty and rinse each waste bottle. The syringe can dispense about 4 ml of waste into the waste bottle or about 500 washes for a 10- μ l syringe.

Figure 2-13



Shows position of syringe tip when dispensing waste.

3. Place the bottles in the appropriate positions on the injector turret according to the table on page 46. The positions are labeled on the turret, Solvent A, Waste A, Solvent B, and Waste B.

Caution.

Do not refill a solvent bottle that still has solvent left in the bottle. The solvent from the last analysis may be contaminated.

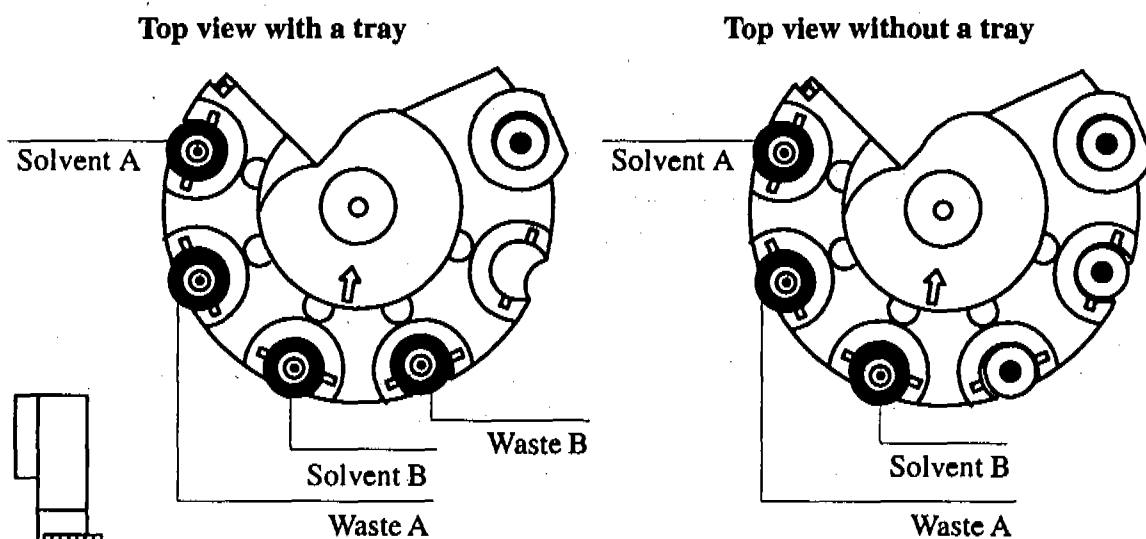
Preparing for Operation

Preparing the solvent and waste bottles

Figure 2-14 shows an example of the turret positions used with and without a tray.

- a. If a tray is installed, place four bottles in turret positions solvent A, waste A, solvent B, and waste B.
- b. If a tray is not installed, place two or three bottles (solvent A, waste A, and solvent B) in the turret positions. Position 3 in the turret can be used as sample 3 or solvent B. Make sure you have the correct vial in the turret.

Figure 2-14



Examples with and without a tray.

Caution

When a tray is installed, place a waste bottle in **both** waste position A and waste position B. The injector alternates dispensing waste between the two positions. With the HP 6890 Series GC, you can choose A, B, or both.

Preparing for Operation
Preparing the solvent and waste bottles

| System | | Solvent | Waste |
|---|----------------|---------------|---------------|
| Standalone | With a tray | A & B | A & B |
| | Without a tray | A | A |
| HP 3365/ HP 5890 | With a tray | A, B, or both | A & B |
| | Without a tray | A, B, or both | A |
| HP 3396/ HP 5890 | With a tray | A, B, or both | A & B |
| | Without a tray | A | A |
| HP 3396/ HP 6890 | With a tray | A, B, or both | A, B, or both |
| | Without a tray | A, B, or both | A |
| HP multitechnique ChemStation/ HP 5890 | With a tray | A, B, or both | A & B |
| | Without a tray | A, B, or both | A |
| HP multitechnique ChemStation/ HP 6890 | With a tray | A, B, or both | A, B, or both |
| | Without a tray | A, B, or both | A |

Do you need to read further?

The volumes of the solvent and waste bottles determine the number of sample vials that you can run at one time. If your application requires more than the maximum number of washes listed in figure 2-15, you must read the next section entitled, "Estimating the maximum number of sample vials." The total number of washes includes all solvent (pre- and post-injection) and sample washes using the default needle depth. This is critical to your solvent supply if solvent prewashes are not part of your solvent needed estimation.

Preparing for Operation
Preparing the solvent and waste bottles

Figure 2-15

| Number of Bottles | Solvent Limit | | Waste Limit | |
|-------------------|---------------|------------|-------------|------------|
| | 5 μ l | 10 μ l | 5 μ l | 10 μ l |
| Two bottles | 1,000 | 500 | 2,000 | 1,000 |
| One bottle | 500 | 250 | 1,000 | 500 |

Note: Wash volume is 0.8 times the syringe volume.

The maximum number of washes (pre- and post-injection).

Caution

Do not exceed the solvent and waste limits of the bottles. If you exceed these limits, sample carryover may affect your analysis.

Example 1 (tray not installed): Your application requires 10 sample washes and 10 solvent washes with a 10- μ l syringe. For three sample vials (five injections per vial), you need 150 solvent washes, and you need to dispense 300 syringe volumes of waste. With this example, you are limited to one bottle for solvent and one bottle for waste.

Figure 2-15 shows that you can have up to 250 washes from one solvent bottle and can dispense up to 500 washes into the waste bottle. You are within the limits and do not have to read any further.

Example 2 (tray installed): Your application requires three sample washes and three solvent washes with a 10- μ l syringe. For 40 sample vials (two injections per vial), you need 240 solvent washes, and you need to dispense 480 syringe volumes of waste. With this example, you are using two bottles for solvent and two bottles for waste.

Figure 2-15 shows that you can have up to 250 washes from each solvent bottle and can dispense up to 1,000 washes into the waste bottles. You are within the limits and do not have to read any further.

Preparing for Operation

Preparing the solvent and waste bottles

Example 2b (tray installed): Your application requires three sample washes and three solvent washes with a 10- μ l syringe. If you had 60 samples, you would need 360 washes from the solvent bottles. You would have to place solvent bottles in both positions and set the run parameters for solvent washes from both positions (e.g., one from solvent A and two from solvent B).

Example 3 (tray installed): Your application requires three sample washes, three solvent A washes, and three solvent B washes with a 10- μ l syringe. For 100 sample vials (two injections per vial), you need 600 solvent washes and need to dispense 1,200 syringe volumes of waste. With this example, you are using two bottles for solvent and two bottles for waste.

Figure 2-15 shows that you can have up to 250 washes from each solvent bottle and can dispense up to 1,000 washes into the waste bottles. You would exceed the solvent and waste capacity of the bottles. Read the next section to estimate the maximum number of sample vials you can run at one time.

Estimating the maximum number of sample vials

This section contains equations and tables for estimating the maximum number of sample vials you can run before you must replace the solvent or empty the waste bottles.

Caution

The number of sample vials given in the equations and tables are estimates. Characteristics of the solvent, such as the evaporation rate and surface tension, may affect the capacity of the bottles.

If you use either the tables or the equations, you must know the following parameters for your application:

- The number of injections per vial.
- The number of solvent washes per pre- and post-injection required from each solvent bottle.

Preparing for Operation

Preparing the solvent and waste bottles

- The number of sample wastes and solvent washes per injection that the injector dispenses into each waste bottle. When the tray is installed, you must use two waste bottles. The injector dispenses the waste equally between the two bottles unless you have specified differently on the HP 6890 Series GC.
- The syringe size, 5 µl or 10 µl.

Using the equation to estimate

1. Substitute the parameters of your application into both equations.
 - If you are using a 5-µl syringe, substitute 0.004 ml/wash for the 0.008 ml/wash in each equation.
 - If a tray is installed or both waste bottles are being used, substitute 8.0 ml of waste for the 4.0 ml of waste in the second equation.
2. Calculate the answers for both equations. Use the smaller of the two answers for the estimate.
 - Equation S estimates the maximum number of vials from the volume of solvent available from the bottle associated with the largest number of washes.

Equation S

$$\text{Maximum Number of Vials} = \frac{2.0 \text{ ml of solvent}}{\left(\frac{0.008 \text{ ml}}{\text{wash}} \right) \times \left(\frac{\text{Number}}{\text{injections/vial}} \right) \times \left(\frac{\text{Largest number solvent washes from a bottle}}{\text{washes from a bottle}} \right)}$$

- Equation W estimates the maximum number of vials from the waste bottle capacity.

Equation W

$$\text{Maximum Number of Vials} = \frac{4.0 \text{ ml of waste}}{\left(\frac{0.008 \text{ ml}}{\text{wash}} \right) \times \left(\frac{\text{Number}}{\text{injections/vial}} \right) \times \left(\frac{\text{Number solvent and sample washes/injection}}{\text{washes/injection}} \right)}$$

Preparing for Operation
Preparing the solvent and waste bottles

Equation method example

Assume a tray is installed and your application parameters are:

- Two injections per vial
 - Three washes from solvent bottle A
 - Two washes from solvent bottle B
 - Two sample washes
 - 10- μ l syringe
1. Substitute the parameters of your application into equations S and W.
S: Maximum number of vials = $2.0 \div (0.008 \times 2 \times 3) = 41$
W: Maximum number of vials = $8.0 \div (0.008 \times 2 \times 7) = 71$
 2. Calculate the answers for both equations. Use the smaller of the two answers, **answer (41)**.

Using the table to estimate

1. Go to the **S** table in figure 2-16. In the left-hand column, find the largest number of solvent washes you need from a solvent bottle. If you use a tray, you can divide the solvent washes between *both* waste bottles.
2. Read across this row to the column with the number of injections you are taking from each vial. The intersection of row and column is the maximum number of vials that you can run from the limiting bottle.
 - When you are using a 5- μ l syringe, multiply the maximum number of vials listed in the figure by 2.

Preparing for Operation
Preparing the solvent and waste bottles

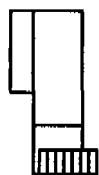
Figure 2-16

S

Number
of
solvent
washes
per
injection

Number of injections per vial

| | 1 | 2 | 3 | 4 | 5 | 6 |
|----|------|------|----|----|----|----|
| 1 | 100+ | 100+ | 83 | 62 | 50 | 41 |
| 2 | 100+ | 62 | 41 | 31 | 25 | 20 |
| 3 | 83 | 41 | 27 | 20 | 16 | 13 |
| 4 | 62 | 31 | 20 | 15 | 12 | 10 |
| 5 | 50 | 25 | 16 | 12 | 10 | 8 |
| 6 | 41 | 20 | 13 | 10 | 8 | 6 |
| 7 | 35 | 17 | 11 | 8 | 7 | 5 |
| 8 | 31 | 15 | 10 | 7 | 6 | 5 |
| 9 | 27 | 13 | 9 | 6 | 5 | 4 |
| 10 | 25 | 12 | 8 | 6 | 5 | 4 |
| 11 | 22 | 11 | 7 | 5 | 4 | 3 |
| 12 | 20 | 10 | 6 | 5 | 4 | 3 |
| 13 | 19 | 9 | 6 | 4 | 3 | 3 |
| 14 | 17 | 8 | 5 | 4 | 3 | 2 |
| 15 | 16 | 8 | 5 | 4 | 3 | 2 |



Maximum number of sample vials with one solvent bottle and a 10- μ l syringe.

3. Go to the **W** table in figure 2-17. In the left-hand column, find the number of solvent wastes and sample washes you need.
4. Read across this row to the column with the number of injections you are making from each vial. The intersection of row and column is the maximum number of vials that you can run with one bottle.
 - If a tray is installed, multiply the maximum number of vials listed in the figure by 2.

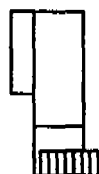
Preparing for Operation
 Preparing the solvent and waste bottles

Figure 2-17

W

| | Number of injections per vial | | | | | |
|----|-------------------------------|------|------|------|-----|----|
| | 1 | 2 | 3 | 4 | 5 | 6 |
| 1 | 100+ | 100+ | 100+ | 100+ | 100 | 83 |
| 2 | 100+ | 100+ | 83 | 62 | 50 | 41 |
| 3 | 100+ | 83 | 55 | 41 | 33 | 27 |
| 4 | 100+ | 62 | 41 | 31 | 25 | 20 |
| 5 | 100 | 50 | 33 | 25 | 20 | 16 |
| 6 | 83 | 41 | 27 | 20 | 16 | 13 |
| 7 | 71 | 35 | 23 | 17 | 14 | 11 |
| 8 | 62 | 31 | 20 | 15 | 12 | 10 |
| 9 | 55 | 27 | 18 | 13 | 11 | 9 |
| 10 | 50 | 25 | 16 | 12 | 10 | 8 |
| 11 | 45 | 22 | 15 | 11 | 9 | 7 |
| 12 | 41 | 20 | 13 | 10 | 8 | 6 |
| 13 | 38 | 19 | 12 | 9 | 7 | 6 |
| 14 | 35 | 17 | 11 | 8 | 7 | 5 |
| 15 | 33 | 16 | 11 | 8 | 6 | 5 |

Number
 of pre- and
 post-solvent
 washes
 +
 sample washes
 per injection



Maximum number of sample vials with one waste bottle and a 10- μ l syringe.

5. Compare the answers from both tables. Use the smaller of the two answers for the estimate.

Table method example

Assume a tray is installed and your application parameters are:

- Two injections per vial
- Three washes from solvent bottle A
- Two washes from solvent bottle B
- Two sample washes
- 10- μ l syringe

Preparing for Operation

Preparing the solvent and waste bottles

1. Go to the **S** table in figure 2-16. In the left-hand column, find the largest number of solvent washes you need from a solvent bottle, **answer (3)**.
2. Read across this row to the column indicating the number of injections you are taking from each vial, **answer (2)**.

The intersection of row and column is the maximum number of vials that you can run from the limiting bottle, **answer (41)**.

3. Go to the **W** table in figure 2-17. In the left-hand column, find the number of solvent and sample washes you need, **answer (7)**.
4. Read across this row to the column indicating the number of injections you are making from each vial, **answer (2)**.

The intersection of row and column is the maximum number of vials for one bottle, **answer (35)**.

- Because a tray is installed, multiply the maximum number of vials listed in the table by 2, **answer (70)**.
5. Compare the answers from both tables. Use the smaller of the two answers for the maximum vial estimate, **answer (41)**.

Controlling sample carryover

This section describes the features of the injector used to control carryover. For an explanation of sample carryover, see the "Glossary."

You can use solvent washes, sample washes, and pumps to control carryover because each dilutes the concentration of sample left in the syringe. The effectiveness of each depends on your application.

You may be able to adjust your application for a more efficient use of solvent and sample and increase the number of sample vials you can run at one time.

Preparing for Operation

Preparing the solvent and waste bottles

- Solvent washes
 - The injector fills the syringe to eight-tenths of its volume (4 μl with the 5- μl syringe and 8 μl with the 10- μl syringe) from either the solvent A or solvent B position. Then it dispenses the syringe contents into one of the waste bottles. Solvent washes can be set to occur before taking a sample (preinjection solvent wash) or immediately after the injection (postinjection solvent wash).
- Sample washes
 - The injector fills the syringe to eight-tenths of its volume with the next sample and dispenses the contents into one of the waste bottles. Sample washes occur before the injection. When sample is limited, you can use a solvent prewash to wet the syringe before drawing sample.
- Pumps
 - The injector fills the syringe to eight-tenths of its volume with the next sample and dispenses it back into the sample vial. Pumps occur after the sample washes and immediately before the injection. Pumps serve to eliminate bubbles. If the needle contains solvent from a previous wash, the pumps may add a small amount of solvent that mixes with the sample and can dilute a small volume.

The number and type of washes are determined with the run parameters. See "Setting the run parameters" in chapter 3 for details.

Under ideal conditions, four washes reduce the carryover to one part in 10,000. The actual number and type of washes you need depends on many factors, including:

- The percentage of carryover that you can accept
- The viscosity and solubility of the analyte(s)
- The volatility of the solvent(s)
- The extent of wear in the syringe barrel

The number and type of washes is often set for you as a standard method. You can also determine the number and type of washes experimentally.

Preparing for Operation
Selecting and installing syringes

To measure the percentage of carryover in your procedure, run a solvent blank after a sample, and compare the peak areas of the components.

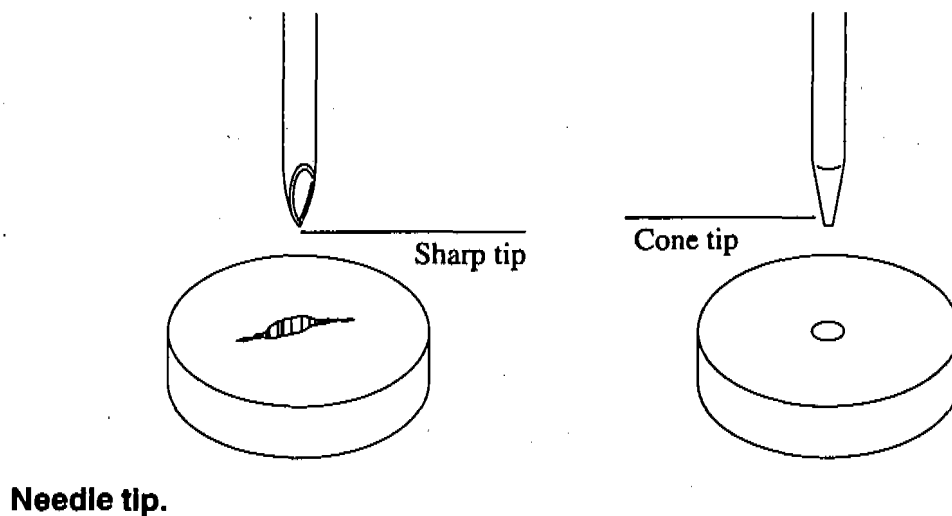
Selecting and installing syringes

Selecting syringes

Select the type of syringe you need based on the inlet (injection port) you are using and the volume of sample you want to inject.

1. The syringe needle must have a cone tip. Do not use sharp-tipped needles. These needles tear the inlet septum causing leaks. Also, sharp-tipped needles wipe off on the septum as they exit resulting in a large solvent tail on the chromatogram.

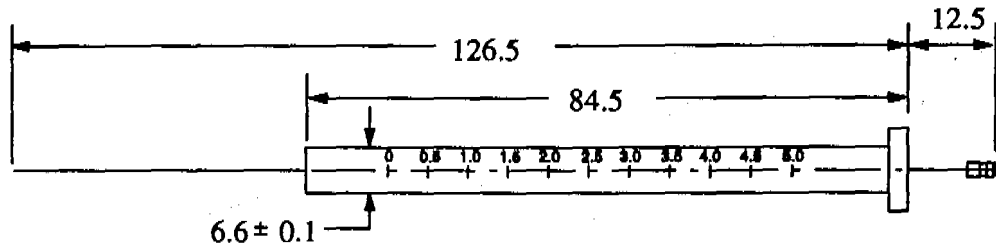
Figure 2-18



Preparing for Operation
Selecting and installing syringes

Figure 2-19 illustrates some of the critical syringe dimensions. These dimensions do not make up a complete set of specifications. Some of the dimensions are too difficult to measure without special instruments.

Figure 2-19

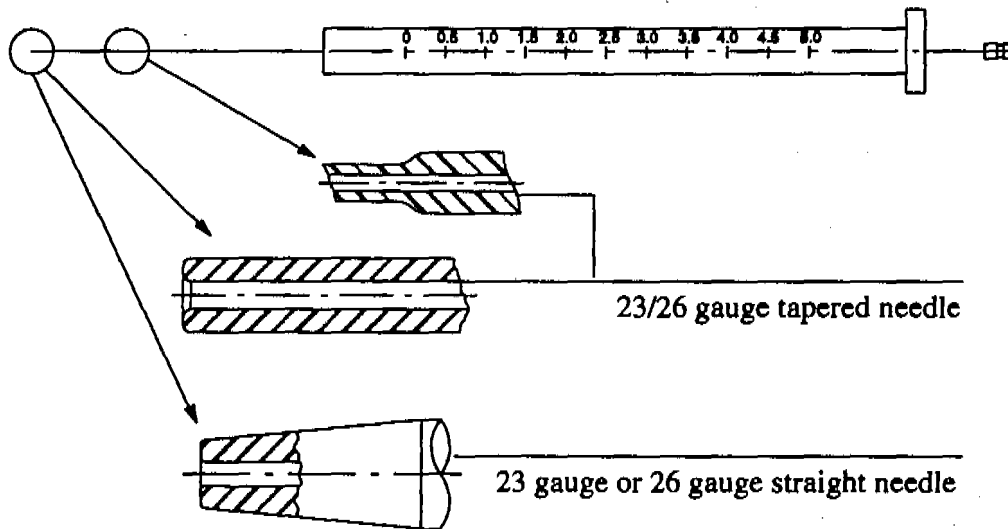


All dimensions in millimeters

Syringe dimensions.

Figure 2-20 illustrates the shapes of the two fixed needles:

Figure 2-20




Needle shapes.

Preparing for Operation
Selecting and installing syringes

2. Select the appropriate syringe needle gauge. If you need more help in making your selection, refer to chapter 6, "Special Topics."

Figure 2-21

| | Inlet | Needle Gauge | Column Type |
|---|-----------------------------|--|----------------------------|
|  | Packed, split, or splitless | 23 gauge or 23/26 gauge tapered | |
| | Cool on-column | 23/26 gauge tapered, 26 gauge, 32/26 gauge | 530 μm 320 μm 250 μm |

Needle gauge selection.

3. Select the 5-μl or 10-μl syringe. Figure 2-22 shows the range of volumes you can inject according to the sample volume setting, the syringe size, and whether the nanoliter adapter, accessory HP 18599N, is installed.

Figure 2-22

| Sample Volume Setting | Syringe Size | Standard Injection | | With Nanoliter Adapter | |
|-----------------------|--------------|--------------------|-------|------------------------|-------|
| | | 5 μl | 10 μl | 5 μl | 10 μl |
| 1 | | 0.5 | 1.0 | 0.1 | 0.2 |
| 2 | | 1.0 | 2.0 | 0.5 | 1.0 |
| 3 | | 1.5 | 3.0 | 1.0 | 2.0 |
| 4 | | 2.0 | 4.0 | 1.5 | 3.0 |
| 5 | | 2.5 | 5.0 | 2.0 | 4.0 |

Injection volumes depending on sample volume setting, syringe size, and injection type.

Caution

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

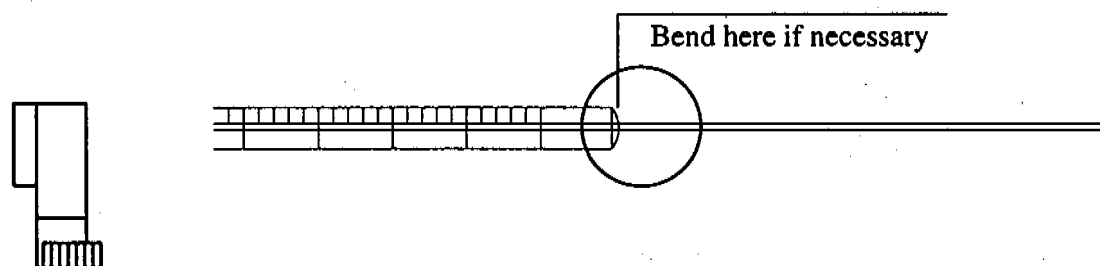
Preparing for Operation
Selecting and installing syringes

Inspecting syringes

Before installing the syringe:

1. Roll the syringe on the edge of a clean flat surface. If the tip of the needle seems to follow a circular motion, bend it slightly near where it connects to the syringe barrel, and check it again.

Figure 2-23



Syringe parts and needle inspection.

2. Check for a rough needle. The needle may contain closely spaced concentric ridges that act like a miniature file and abrade pieces of the septum into the inlet. The ridges are easy to see under 10X magnification.

If there are ridges, polish the needle by pulling it through a folded piece of fine emery paper between your finger and thumb until the ridges are gone. Be careful not to modify the special blunt tip of the syringe.

3. Check for a sticky plunger. Slide the plunger of the syringe up and down a few times. It should move smoothly—without sticking or binding. If it is sticky, remove the plunger, and clean it with solvent.

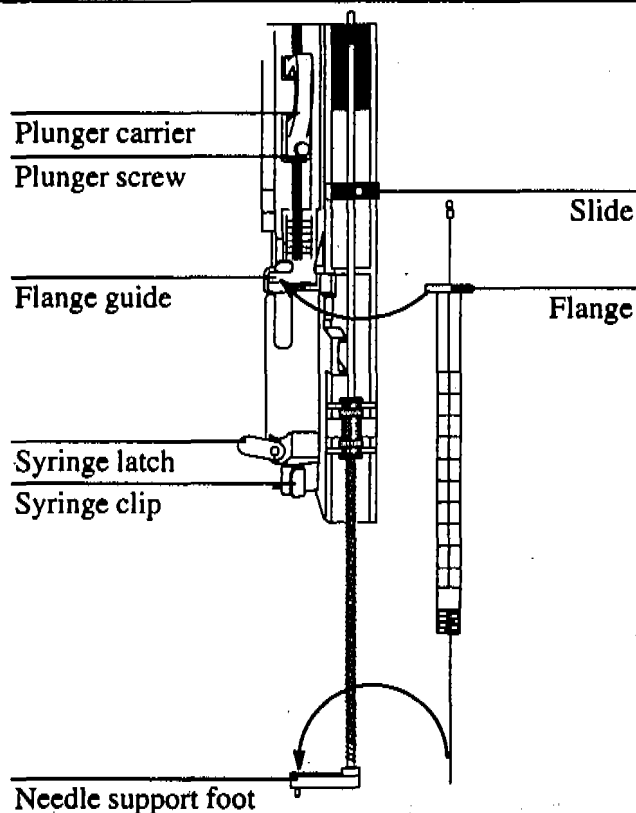
Preparing for Operation
Selecting and installing syringes

Installing syringes

After completing this task, check your work with the following instructions:

1. Open the injector door.
2. Pass the syringe needle through the hole in the needle support foot.
3. Align the syringe barrel with the flange guide and syringe clip, and press the syringe into place, keeping the needle in the hole of the needle support foot.
4. Close the syringe latch by swinging it clockwise.

Figure 2-24



Installing the syringe in the syringe carriage and needle support.

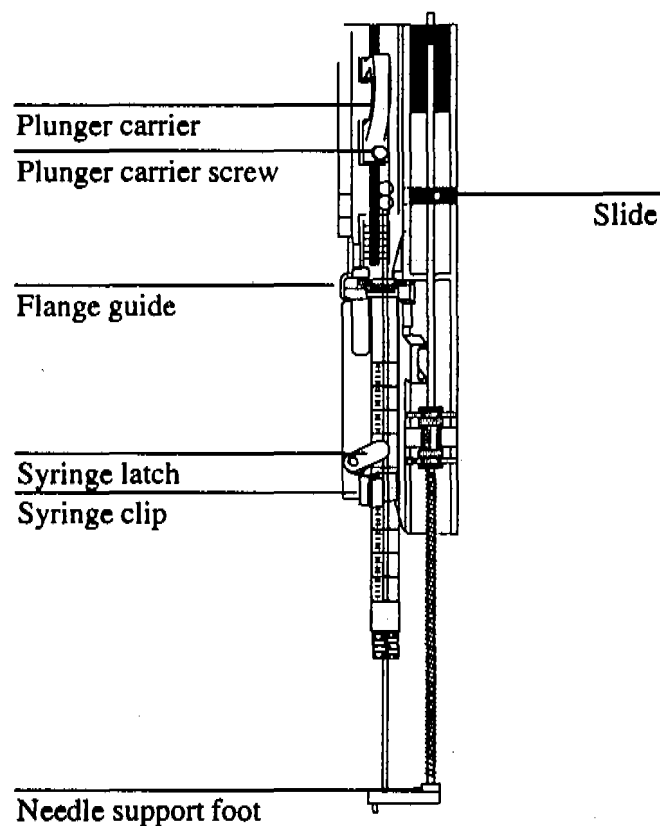
5. Move the plunger carrier loop down, and tighten the plunger screw.

Preparing for Operation
Selecting and installing syringes

Checking your work

- Move the plunger carrier up and down. If the syringe plunger does not move along with the carrier, repeat the previous steps. Be sure the plunger carrier screw is tight.
- Check that the needle is aligned with the needle guide in the foot by moving the slide up and down. The needle should slide smoothly in the needle guide.

Figure 2-25



Plunger carrier and needle support with needle installed.

Caution

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

Preparing for Operation

Maintaining the Inlet

Caution

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

- To check the alignment of the syringe needle to ensure an average septum life of 200 injections, follow these instructions:
 1. Pull down the syringe carriage until the needle tip is near the top of the inlet septum nut. Figure 2-26 shows a syringe needle over the septum retainer nut of a packed inlet with the turret removed for a better view.

The needle should be centered exactly over the hole in the septum retainer nut. Use a small light to see that the needle will hit the septum without rubbing on the nut.
 2. If it is not centered, check that the syringe is installed correctly in the syringe carriage, the syringe needle is straight, and the mounting bracket is aligned with the inlet.

Maintaining the inlet

This section contains inlet information to help you maintain good chemical performance from your GC.

Changing septa

The average life of an inlet septum should be 200 injections. This is influenced by the alignment of the injector bracket, the syringe, and the type of septum.

When you replace the septum:

- Clean off the metal surfaces that create the seal.
- Replace with the correct septum.
- Make sure the bottom of the septum is flat against the base.

Preparing for Operation

Maintaining the Inlet

- For packed and split/splitless inlets, tighten the septum retainer nut only until the split ring begins to turn on the top of the nut.

Caution

Do not tighten the septum retainer nut too much. The syringe needle will core the septum and push out pieces of the septum material into the inlet. Septum life is often reduced to as few as five injections.

Changing or cleaning liners

Most samples contain nonvolatile material that accumulates on and partially pyrolyzes on the liner. Replace the liner or clean the liner periodically.

Some active components interact with the glass liner and glass wool plug of the capillary liner. If these components are in the sample, the liner should be deactivated before installation and periodically replaced or cleaned and deactivated. There are several commercially available solutions for deactivating the liner. Take the appropriate safety precautions when using any of these solutions. Refer to the *Analytical Columns and Supplies Catalog* for deactivated liners.

Suggestions for packed inlets with 530- μm columns

- When using a heated, packed inlet with a 530- μm column, do the following:
 - Install the column so that no more than 1 to 2 mm of the column extends past the ferrule. This avoids large unswept volumes at the base of the inlet.
 - Use polyimide ferrules (Vespel) instead of graphite. A small portion of the column ferrule is exposed to sample vapor.
 - Insulate the part of the inlet that projects into the oven. If the oven is programmed to increase temperature, the lower part of the inlet could become a cold spot.

Adapting for cool on-column injection

For cool on-column inlets, you must specify on-column injection with the sampler parameters so that the injector:

- Slows the carriage speed to 500 milliseconds. The syringe needle moves more slowly into and out of the inlet and spends more time in the inlet.
- Lowers the tip of the syringe needle an additional 19 mm (3/4-inch) to the column.

You can use the slow injection parameter along with on-column injection. This adds the following change to the injection:

- Slows the plunger speed to 1/18 the normal speed

Complete the following:

1. Install an on-column syringe. Hewlett-Packard recommends syringes with a 23/26 gauge tapered-tip needle for injections onto 530- μ columns and 32/26 gauge for 320- and 250- μ columns.
2. If the injector is controlled by an external device, such as an integrator or a workstation, change the on-column parameter to ON or YES.

If the injector is controlled by an HP 6890, this will be set automatically when the GC configures for the on-column inlet.

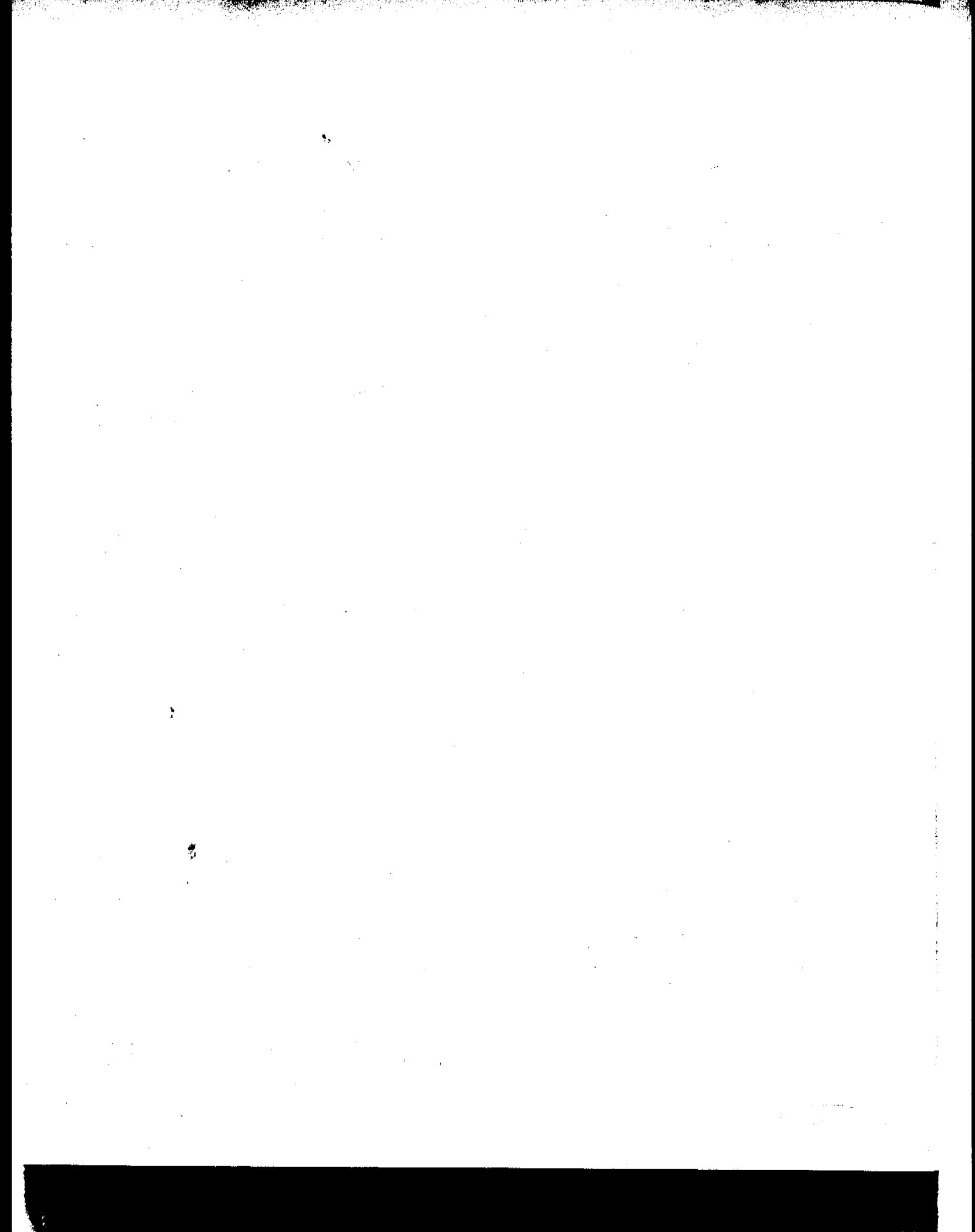
If the injector is operated with standalone control, change the on-column switch to the YES, or down, position. For more information on the on-column switch, see "On-column injection mode" in chapter 4.

3. The inlet must be configured for cool on-column injection. Install the appropriate needle guide, septum, spring, and insert for the GC.

If you have an on-column inlet on an HP 5890A with a cooling tower (HP part no. 19245-20540), replace it with a septum nut base assembly (HP part no. 19245-80520).

If you are performing automated injection onto 320- μ m and 250- μ m columns, replace the standard septum nut base assembly (HP part no. 19245-80520) with the small septum nut base assembly (HP part no. 07673-61330)

4. For injecting onto 320- μ m or 250- μ m columns, see "Performing cool on-column injection onto 250- μ m and 320- μ m columns" in chapter 6.



Operation

Operation

This chapter contains detailed information for operating the automatic liquid sampler, including:

- Setting the run parameters
- Starting and stopping the automatic liquid sampler
- What happens during a run
- Running the samples
- Four methods of control
- Using two injectors
- Examples of operation

Setting the run parameters

This section describes each run parameter, lists the range of values for the common controlling devices, and contains three examples.

The run parameters tell the injector and the tray what to do. For example, they tell the injector how much sample to inject and tell the tray where to find sample vials. You can set many of the parameters either with the control switches of the injector or with an external device, such as an integrator, ChemStation, or the HP 6890 Series GC.

For information about setting parameters with the control switches on the injector panel, see chapter 4, "Standalone Control."

Description of the parameters

Injections per vial

Specifies the number of times the run is repeated for each sample vial. For example, if the value is two, each vial is analyzed twice.

Caution

Too many injections per vial can allow sample to evaporate, deplete sample, or contaminate sample in the vial. This may affect the reproducibility of the injection volume.

Position of first vial

Specifies the tray or injector turret position of the first sample vial. Each position on the tray and turret is numbered. Tray values range from 1 to 100. Only three sample positions are available in the standard injector turret.

Position of last vial

Specifies the tray or injector turret position of the last sample vial. Tray values range from 1 to 100. Only three sample positions are available in the standard injector turret.

Number of sample washes

Specifies the number of times the syringe is rinsed with sample before the injection. The injector lowers the needle into the sample vial, fills the syringe to eight-tenths its full volume, and empties it into one of the waste bottles.

Number of sample pumps

Specifies the number of times the syringe plunger is moved up and down while the needle is in the sample to expel air bubbles and improve reproducibility.

Viscosity delay

Specifies the number of seconds the plunger pauses at the top of the stroke during sample pump, sample draw, and sample and solvent washes. For viscous samples, the pause during sample draw allows the sample to flow into the vacuum created by the syringe. During wash strokes, it allows diffusion of the viscous sample into the wash solvent. Viscosity delay settings range from 0–7 seconds.

Solvent washes—pre- and post-injection

Number of solvent A washes:

Specifies the number of times the syringe is rinsed with solvent from the solvent A bottle. The injector lowers the needle into the solvent A bottle, fills the syringe to eight-tenths its full volume, and empties it into one of the waste bottles.

Number of solvent B washes:

Specifies the number of times the syringe is rinsed with solvent from the solvent B bottle. The injector lowers the needle into the solvent B bottle, fills the syringe to eight-tenths its full volume, and empties it into one of the waste bottles.

Operation

Setting the run parameters

Volume

Specifies the injection volume. Each volume stop corresponds to a volume of one-tenth the syringe's capacity. Valid setpoints are 1–5 stops.

Caution

Do not set the volume parameter for splitless inlets more than 2 μl . Larger volumes cause sample to be lost through the purge vent and may contaminate carrier inlet lines.

Dwell time

This setpoint specifies the time delay of the needle in the inlet for both pre- and post-injection. This adds the following changes to the injection parameters:

- Preinjection dwell: Time delay of the needle in the inlet before the plunger is depressed to inject sample. When preinjection dwell is selected, one stop of air is drawn after the sample for volume settings from 1–4 stops. With a setpoint of five stops, no air is drawn.
- Postinjection dwell: Time delay after injection before needle is withdrawn from the inlet

The allowable range for both dwell times is 0–1 minute in increments of 0.01 minute.

Priority samples

This setpoint specifies whether the arm of the tray should look for a priority sample in position 100 between runs. If the feature is turned on, the tray checks position 100 before each regular vial in the sequence. If a vial is in position 100, the tray arm delivers the vial to the injector turret. After the injection, the tray arm places the vial in position 99.

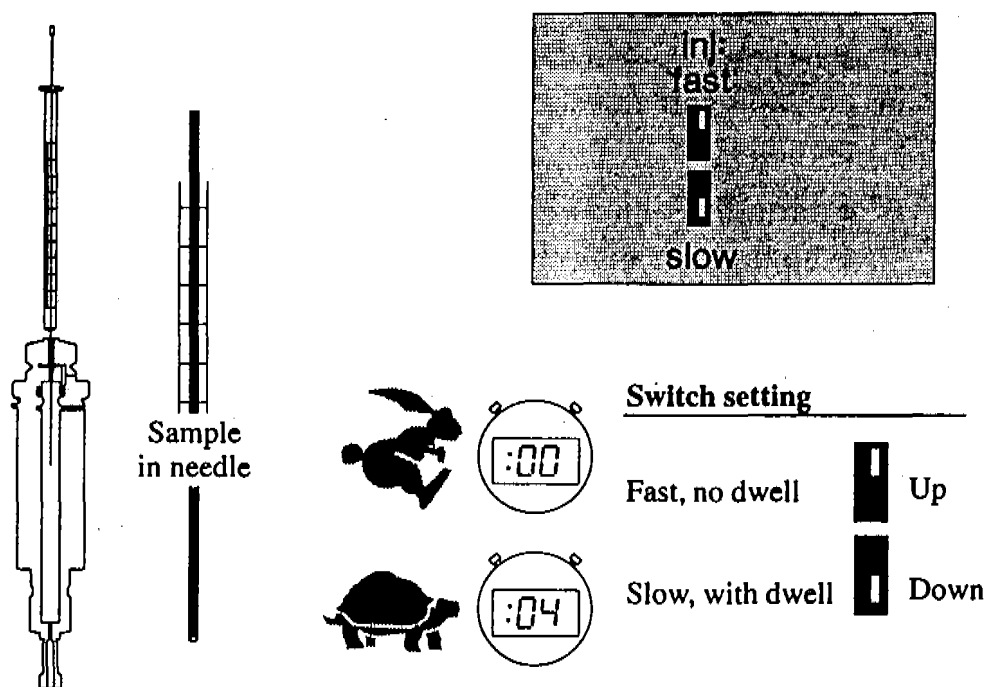
If you are using the priority sample feature, use only tray positions 1 to 98.

Operation
Setting the run parameters

Slow injection

This setpoint specifies the speed of the syringe plunger during the injection stroke and whether or not the syringe needle stays in the inlet (dwell time) after injection of the sample. It enables you to reduce the average speed of the plunger from about 100 $\mu\text{l}/\text{sec}$ to about 5 $\mu\text{l}/\text{sec}$ (with a 10- μl syringe). This parameter also holds the syringe in the inlet for 4 seconds after the injection. The plunger speed during the pump and waste dispensing does not change.

Figure 3-1



Injection speed.

Caution

The slow setting is recommended only for a few specific splitless and cool on-column applications. For most applications, set this switch to "fast." A setting of "slow" for other applications may cause peak area discrimination and reduced quantitative precision.

Operation
Setting the run parameters

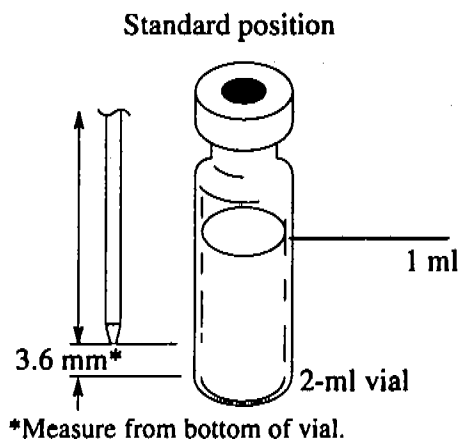
Slow plunger

This setpoint specifies the speed of the syringe plunger during injection. It enables you to reduce the average plunger speed. The plunger speed during the pump and waste dispensing does not change. Selecting slow plunger reduces the rate to about 5 $\mu\text{l}/\text{sec}$, compared to the fast speed of about 100 $\mu\text{l}/\text{sec}$ (with a 10- μl syringe).

Sampling depth

This setpoint allows you to move the position of the needle tip up or down to approximate locations from the nominal position of zero (default). Default is the standard position (see figure 3-2). Default is the standard position (see figure 3-2).

Figure 3-2



Variable sampling depth.

Injection mode

Specifies whether the injector is in the normal or on-column injection mode. The normal injection mode is used with packed and split/splitless inlets. The on-column injection mode is used with cool on-column inlets. For the on-column injection mode, the injector reduces the carriage speed and lowers the tip of the syringe needle an additional 19 mm (3/4-inch) into the column.

Setting the run parameters

Caution Be sure you are using the correct injection mode. If you use the wrong injection mode, you may damage the column, needle, and syringe.

For more information on setting up the inlet for on-column injection, see Chapter 4 of the *HP 6890 Series Gas Chromatograph Operating Manual* (HP part no. G1530-90310) or the *HP 5890 Series II and Series II Plus Programmable Cool On-Column Manual* (HP part no. 19245-90300).

Position

This setpoint indicates which tower is on which inlet (front or back). Typically, the location of the injector cable in the controller box, front or back, is the position associated with that injector.

With an INET integrator and dual injectors, this setpoint specifies which injector, front or back, is associated with the INET data channel and which is associated with the auxiliary data channel. This parameter determines where the sample vial numbers are printed when more than one injector is installed.

On the HP 6890 Series GC, the position of the injector cables are not significant because you can set the injector position from the keypad. With only one injector, you no longer have to move the cables when you move the tower, merely reconfigure the tower position.

Caution Always turn off the power to the controller before you remove and reconnect the injector cables.

Operation
Setting the run parameters

Range of values for common controlling devices

Figure 3-3

| | HP 6890 and HP Multitechnique ChemStation* | INET HP 3396 Integrator | HP 3365 ChemStation** |
|--|---|--|-------------------------------------|
| Injections per vial | 1-99 | 0-15 | 1-99 |
| Position of first vial (front) | 1-3, 101-103, or 1-100 | 1-3 or 1-100 | 1-3 or 1-100 |
| Position of first vial (back) | 1-3, 201-203, or 1-100 | 1-3 or 1-100 | 4-6 or 1-100 |
| Position of last vial (front) | 1-3, 101-103, or 1-100 | 1-3 or 1-100 | 1-3 or 1-100 |
| Position of last vial (back) | 1-3, 201-203, or 1-100 | 1-3 or 1-100 | 4-6 or 1-100 |
| Number of sample prewashes | 0-15 | 0-15 | 0-15 |
| Number of pumps | 0-15 | 0-15 | 0-15 |
| Variable sampling depth | -2-30 mm | n/a | n/a |
| Viscosity | 0-7 sec | 0-7 sec | 0-7 sec |
| Volume (Stops) (tenths of syringe volume) | 1-5 | 1-5 | 1-5 |
| Number of solvent A washes (post) | 0-15 | 0-15 | 0-15 |
| Number of solvent A prewashes | 0-15 | 0-15 | 0-15 |
| Number of solvent B washes (post) | 0-15 | 0-15 | 0-15 |
| Number of solvent B prewashes | 0-15 | 0-15 | 0-15 |
| Preinjection dwell | 0-1 min | n/a | n/a |
| Postinjection dwell | 0-1 min | n/a | n/a |
| Priority samples | Create sequence | 0 = no, 1 = yes | Create special method |
| On-column injection mode | Set by GC | | |
| Normal mode | | 0 | No |
| On-column mode | | 1 | Yes |
| Variable plunger speed | Yes/No | | |
| Fast | | 1 | 1 |
| Slow | | 0 | 0 |
| Position | 1 = front, 2 = back | | Set during instrument definition |

* MS Chemstation rev 3.71 and above

**MS Chemstation rev 3.65 and below

Range of parameter values for common control devices.

What happens during a run?

In the following description, the run parameters that determine the automatic liquid sampler's actions are surrounded by quotes ("run parameter"). From the point when you start the run, the automatic liquid sampler does the following:

1. The syringe carriage, plunger carrier, injector turret, tray arm, and gripper move to their home positions.
2. With a tray, the tray gripper picks up a vial from the "position of first vial" and delivers it to the injector turret associated with sample "position." Without a tray, the samples are already in the injector turret.
3. If this is the first sequence after the power is turned on, the injector goes through a solvent "prewash" and pauses for "viscosity delay."
4. The injector lowers the syringe into the vial ("sampling depth"). The injector fills the syringe with sample, pauses for "viscosity delay," and dispenses it into one of the waste bottles according to the "number of sample washes."
5. The injector fills the syringe with sample and dispenses it back into the sample vial according to the "number of pumps." The syringe plunger may pause at the top of the pump stroke according to the "viscosity delay."
6. The injector fills the syringe with the "volume/stop" of sample. The syringe plunger may pause at the top of the injection stroke according to the "viscosity" then draws up one stop of air for "predwell." The injector then lowers the syringe into the injection port, remains for the "preinjection dwell," injects it, applying "plunger speed", and holds it for the "postinjection dwell time."
7. The injector fills the syringe with solvent from solvent bottle A, pauses for "viscous delay" with the syringe over the waste bottle, and dispenses it into one of the waste bottles according to the "number of solvent A washes."

Operation

What happens during a run?

8. The injector fills the syringe with solvent from solvent bottle B, pauses for "viscosity delay" with the syringe over the waste bottle, and dispenses it into the same waste bottle according to the "number of solvent B washes."
9. With a tray, the tray gripper picks up the vial and returns it to its original position in one of the tray quadrants.

The injector waits until the GC sends a ready signal to continue. Steps 1, 2, and 3 through 9 are repeated according to the "injections per vial."

With a tray, the steps are repeated until the tray returns the last sample to the "position of last vial." Without a tray, the steps are repeated according to the rotary switch setting.

If the "priority sample" feature is turned on, the tray gripper looks for a sample in tray quadrant position 100 after each vial. After a priority sample is run, the tray returns it to tray quadrant position 99.

Checklist

Use this checklist to make sure the sampler is ready before you begin.



- Sample vials are half full
- Cap centered, no wrinkles, septum is flat
- Sample inserts and vials match the run parameters
- Tray quadrants snapped in place



- 4.5 ml of fresh solvent in each solvent bottle
- Waste bottles are clean and empty
- Two waste bottles (with tray)
- Limit to number of sample vials



- New or clean syringe
- Correct syringe design and size
- Plunger carrier holds plunger button
- Needle is aligned with septum retainer nut
- Syringe rinsed with solvent



- Liner is clean and deactivated
- Liner is correct type for injection technique
- Septum type is correct
- Septum is less than 200 injections old
- Correct septum nut



- Injector run parameters are set correctly
 - Injection mode matches type of inlet
 - Number of injections per vial is less than five
 - Injection volume is not greater than 2 μ l for splitless inlet

Running the samples

This section describes starting and stopping a run or sequence and the movements of the automatic liquid sampler during a run.

Starting a run or sequence

After completing the checklist on the previous page, start the run or sequence by pressing the start button on the injector or entering the appropriate command at your controlling device.

Stopping or interrupting a run or sequence

The following events interrupt a run:

1. The power to the controller or controlling device fails.
2. You press the stop button or Stop/Abort from a ChemStation.
3. The sampler recognizes a safety or operator fault, such as:
 - You open the syringe access door.
 - The tray gripper drops a sample vial.
 - The syringe needle hits the metal side of the crimp cap.

The sampler responds to these interruptions differently depending on the controlling device. With standalone control, any of the interruptions causes the automatic liquid sampler to stop and abort a sequence.

With the HP 3396 integrator and INET communications, power failures only interrupt the sequence. The integrator recovers and restarts the sequence with the last sample run on the automatic liquid sampler. If the interruption is caused by number 2 or 3, the run is aborted, and the sequence must be restarted.

If the interruption was caused by a problem that the sampler recognizes, it provides you with some feedback by a combination of flashing LED indicators or error messages or a ChemStation message. For more information, see chapter 5, "Preventive Maintenance and Troubleshooting."

Operation Using two Injectors

To restart an aborted sequence from the point of interruption:

1. Identify the last sample vial that was run successfully. Check the vial number of the last successful chromatogram.
2. Reset the parameter that identifies the first sample vial to the next vial number.
3. Press the stop button to clear any faults.
4. Start the revised sequence from your controlling device. From the HP multitechnique ChemStation, you can run a partial sequence to keep the original sample numbers.

Using two injectors

Two-injector configurations have the following characteristics:

- Input and output signals are shared.
- The system sends only one **injector ready** signal to the GC when both injectors are ready and one **start** signal at the beginning of the injection stroke.
- The injectors can operate in the synchronous mode (both inject simultaneously).

The capabilities of a two-injector configuration vary with the communication device.

For information on using two injectors with standalone control or for operation in asynchronous standalone mode, see chapter 4, "Standalone Control."

With a tray

The tray delivers sample vials to the front and back injectors according to the run parameters. There is a separate set of run parameters for each injector.

The tray delivers vials to the front injector before the back injector.

Operation

Using two Injectors

When you set up the run parameters, you must identify which data channel is for which injector.

Different communication devices assign the position and data channel differently.

- Using an HP 6890 Series GC, the channel is assigned when you set up the sequence.
- Using the HP ChemStations, you assign the channel when defining the instruments.
- Using the HP 3396 integrator (INET), you assign the channel when you set up the run parameters for the second injector.

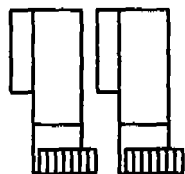
Once both injectors have samples, they begin the injection cycle, including any sample washes. After both injectors have completed the specified sampling steps, they inject the samples. After the injection, both injectors go through the solvent wash cycle. When both injectors are done, then the tray picks up the sample vial from the front injector first and returns it to its original position on the tray, then picks up the vial from the back injector and returns it to its original position.

When the parameters tell the tray arm to deliver the same sample vial to both injectors at the same time, the tray delivers the vial to the front injector first. After the injection, the tray arm delivers this sample to the back injector and delivers the next sample vial in the front injector sequence to the front injector. These samples are injected at the same time. Similarly, if the first and last sample vial parameters are the same for both injectors, the samples are injected in the synchronous N-1 injection mode (see figure 3-4).

Note: The ChemStation method for a single run will not allow you to set the same vial number for front and back injector, however, you can run a dual-column sequence with a single set of vials.

Operation
Using two Injectors

Figure 3-4



| Injection number | Front Injector | Back Injector |
|------------------|----------------|---------------|
| 1st | Vial 1 | Idle |
| 2nd | Vial 2 | Vial 1 |
| 3rd | Vial 3 | Vial 2 |
| . | . | . |
| . | . | . |
| . | . | . |
| nth | Vial n | Vial n-1 |
| Last | Idle | Vial n |

Synchronous N-1 Injection mode.

To inject the same sample into the front and back inlet in the same run, you must do the following:

1. Prepare two sets of sample vials, two vials for each sample.
2. Place the sets in two different locations in the quadrants.
3. Set up the sequence parameters so that the tray delivers the first bottle of one set to the front injector and the first bottle of the other set to the back injector.

For example, with ten samples, you must have 20 vials (two vials for each sample). Place one vial of each in tray positions 1 to 10. Place one vial of each in tray positions 11 to 20. Set the first vial position for the front injector to 1 and the last vial position to 10. Set the first vial position for the back injector to 11 and the last vial position to 20.

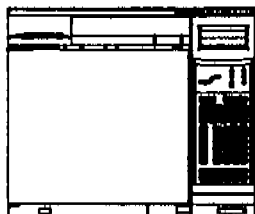
Without a tray

The injectors go through the same motions as with a tray. The injectors inject samples from position 1 first, followed by samples from positions 2 and 3. If the two injectors do not have the same number of samples, the injector that completes its sequence first remains idle while the other finishes.

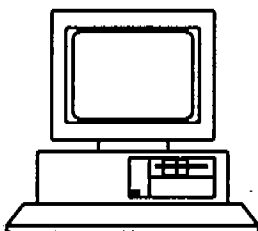
You can mount two injectors onto the HP 6890, the HP 5890, and the HP 5880 GCs. You cannot mount two injectors onto HP 5700 Series GCs.

Four methods of control

There are a number of ways to control the automatic liquid sampler. Each controlling device allows different functionality when using the HP G1513A or HP 18593B injectors. Always check the manual of your chosen controlling device for exact feature sets.



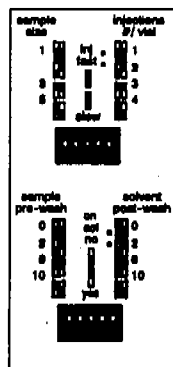
HP 6890 Series GC control.



The HP multit technique ChemStation control with an HP 5890 GC or HP 6890 Series GC, the HP 3365 ChemStation control (MS-DOS series) with an HP 5890 GC, or the MS ChemStation.



An HP 3396 integrator control using the Instrument Network (INET) with an HP 5890 GC or HP 6890 Series GC.



Standalone control with an HP 5890 or an HP 5700 Series GC.

Examples of operation

This section describes the basic steps to set up your automatic liquid sampler using different methods of control. The following examples are for three specific systems with one injector module mounted over the front inlet. For more information on other types of configurations, see the manual of your controlling device.

- HP 6890 Series GC
- HP 3396 integrator with an HP 5890 GC
- HP multitechnique ChemStation with an HP 6890 Series GC
- HP 3365 ChemStation (MS-DOS Series) with an HP 5890 GC

For information on controlling the automatic liquid sampler without an external instrument, see chapter 4, "Standalone Control."

To complete the following examples successfully, the following statements should be true:

- The automatic liquid sampler is installed correctly on the GC.
- The external controlling device is installed and configured correctly.
- You have a basic understanding of the GC and the external controlling device.

Preparing the GC and controlling device

For a trial run, you can minimize the time for the GC to be ready by setting the oven to a low temperature, removing any temperature ramp program, and setting the equilibration time to zero. You can minimize the amount of time the integrator or computer collects data by turning the detector or signal off and setting the run time to about 10 seconds.

To set up your automatic liquid sampler for a run, the following examples will help you get started.

HP 6890 Series GC Control

The following procedure allows you to configure the setpoints associated with the injector waste bottle and tower positions.

Configure tower position

Injector cables are connected to either the INJ1 (front) or INJ2 (back) port on the controller. This setpoint indicates which tower is on which inlet. With only one injector, you no longer have to move cables when you move the tower, merely reconfigure the tower position.

Configure waste bottle position with a tray

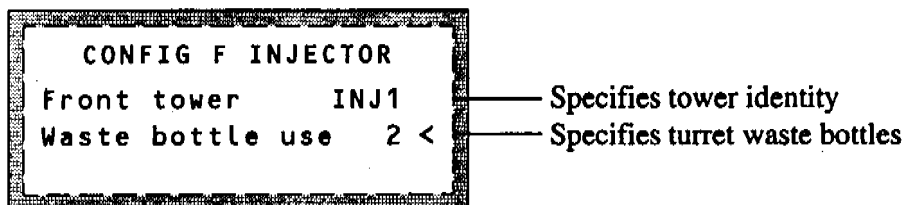
The turret waste bottle positions are controlled by entering a value of:

- 0 for the A position only
- 1 for the B position only
- 2 to alternate between the two waste bottles

Configure the injector

To configure the injector:

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



2. With the cursor on a tower line, use the [On] or [Off] key to set the tower position to either INJ1 or INJ2.
3. Enter a value for waste bottle use.

When the tray is disabled, only waste A is used.

Injector parameter setpoints

To enter injector setpoints:

1. Press [Front Injector] or [Back Injector].
2. Scroll to the desired setpoint.
3. Enter a setpoint value, or turn the setpoint on or off.

| FRONT INJECTOR | |
|--------------------|------|
| Injection vol | 1< |
| #Sample pumps | 1 |
| Viscosity delay | 0 |
| #Sample washes | 1 |
| #Solv. A washes | 1 |
| #Solv. B washes | 0 |
| Stop plungers | Off |
| Pre dwell time | 0.00 |
| Post dwell | 0.00 |
| Sampling offset | On |
| Offset | 2.0 |
| # Solv. A pre wash | 2 |
| # Solv. B pre wash | 0 |

*The Offset line appears only when "On" is selected.

Sample tray setpoints

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

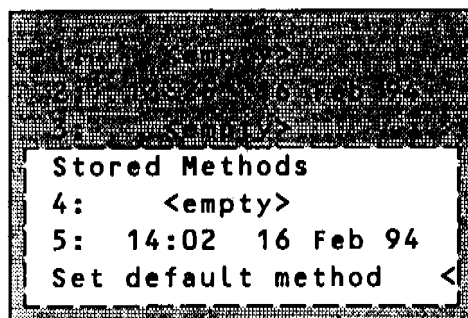
| SAMPLE TRAY | |
|-----------------|-----|
| Enable tray | On |
| Enable bar code | Off |

2. Press [On] or [Off] to enable or disable the tray.
3. Press [On] or [Off] to enable or disable the bar code reader.

Storing injector setpoints

After setting up injector setpoints, sample tray setpoints, and bar code reader configurations, store them as part of a method. This method becomes a part of the sequence used to run the samples.

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



2. Press the [Store] key. Press [Enter] to confirm the store.

Operating in cool on-column mode

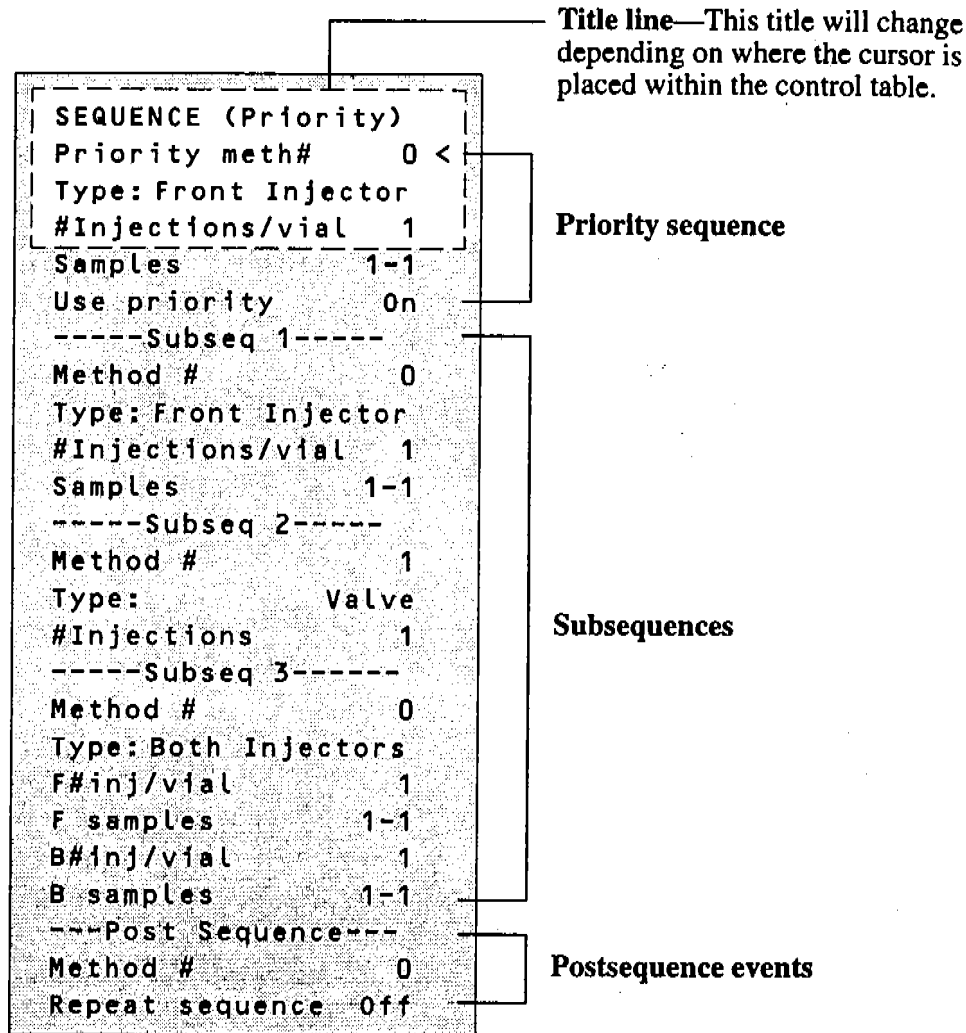
When the injector is configured to a cool on-column inlet, the on-column mode is selected automatically.

Creating a sequence

A sequence specifies which samples to run and the stored method to be used for each. It is divided into subsequences, each of which uses a single method plus a priority sequence and postsequence events.

The sequence definition control table is accessed by pressing [Seq]. The [Seq] key toggles between the stored sequence control table and the sequence definition control table (figure 3-5).

Figure 3-5



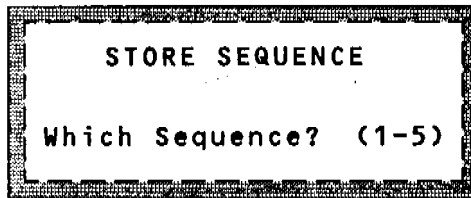
Sequence definition control table.

When in the sequence control table, you will find the [Info] key useful if an explanation of sequence parameters is needed.

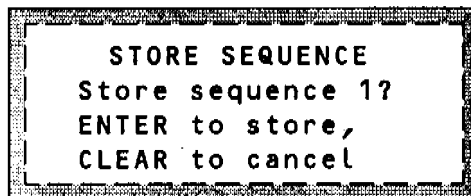
Storing a sequence

Up to five sequences can be stored.

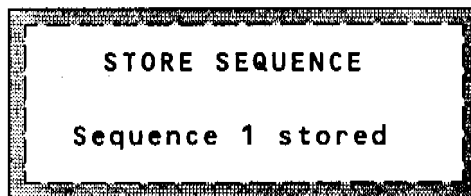
1. To store a sequence, press [Store][Seq]. This opens the Store Sequence control table.



2. Enter an identifying number for the sequence.



3. Press [Enter] to store the sequence.



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

- Overwrite the existing sequence, which will replace the existing sequence with the new sequence.

```
STORE SEQUENCE
Sequence 2 exists,
ENTER to overwrite,
CLEAR to cancel
```

- Cancel the store, and return to the STORED SEQUENCES status table.

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

Starting/running a sequence

Press the [Seq Control] key, scroll to the start sequence line, and press [Enter].

```
SEQUENCE CONTROL
Status:      Stopped
Start sequence <
```

Pressing [Enter] changes the sequence status to Running.

```
SEQUENCE CONTROL
Status:      Running
Pause sequence <
Stop sequence
```

If a sequence is started and the instrument is not ready (due to oven temperature, equilibration times, etc), the sequence will not start until all instrument setpoints are ready.

Special considerations when using an integrator with an HP 6890 Series GC

The definitions of sequence are not the same in the HP 6890 Series GC and in the HP 3396 integrator. The following points must be considered when sequences are used with this GC/integrator combination:

- The integrator has only one subsequence plus the priority sequence. Only one GC method can be used within an integrator sequence.
- The automatic liquid sampler method parameters are prepared using the [Front Injector] and [Back Injector] keys on the HP 6890 keyboard.
- The sample information table is prepared on the integrator.
- The injection sequence parameters can be prepared either with the [Seq] key on the HP 6890 keyboard or in the [PREP][SEQ] dialog of the HP 3396. Setting sequence parameters on either instrument changes the sequence in both places.
- The Start sequence function on the GC is inactive.
- A sequence must be started from the integrator using [SEQ][START].
- If you use INET, pressing the stop key on either instrument stops the run and aborts the sequence.
- If you use the HP part no. 03396-61010 cable instead of INET, the two stop keys have different effects. The [STOP] key on the integrator stops the current run and aborts the sequence. Stop on the GC stops the current run, but the sequence continues as soon as the GC becomes ready.

For more information on the HP 6890 Series GC, see "Using Sequences" in the *HP 6890 Series Gas Chromatograph Operating Manual* (HP part no. G1530-90310).

Figure 3-6 shows an example of the injector parameter portion of a method printout using an HP 6890 Series GC, an HP 3396 integrator, and an HP G1513A injector in the front position and an HP 18593B in the back position.

Figure 3-6



```
* LIST: METH @

INJECTOR 1
Injection vol           1
#Sample pumps          5
Viscosity delay        0
#Sample washes         1
#Solv A washes         3
#Solv B washes         3
Slow plunger           Off
Pre dwell time         0.00
Post dwell              0.00
Sampling offset        On
Offset                 0.0
#Solv A pre wash       0
#Solv B pre wash       0
Front tower            INJ1
Waste bottle use       2

*

INJECTOR 2
Injection vol           1
#Sample pumps          6
Viscosity delay        0
#Sample washes         0
#Solv A washes         0
#Solv B washes         0
Slow injection         Off
Back tower             INJ2
Waste bottle use       2
```

Injector parameters on an HP 3396 Integrator with an HP 6890 GC.

HP 3396 integrator with an HP 5890 GC

1. Set the run time to 0.1 minutes by pressing **TIME** **▣** **1** **STOP**.
2. Begin the dialog to prepare a sequence by pressing the keys for **[PREP][SEQ]**.
3. Answer yes to INET SAMPLER CONTROL by pressing **Y** **ENTER**.
4. Select INJ1 by pressing **ENTER**.

Figure 3-7



```
* TIME .1 STOP
* PREP SEQ

ALS INFORMATION
INET SAMPLER CONTROL [Y*/N]: Y @

7673C SAMPLER:
LOOP ADDRESS:      8

ENABLE TRAY (1=YES)      1 ->

FRONT INJECTOR
INJ/BOTTLE           3 ->
```

HP 3396 Integrator with an HP 5890 GC.

5. Set the number of injections per sample vial to one.

INJ/BOTTLE 1 --> Press **1** **ENTER**

6. Identify the location of the first sample vial.

FIRST BOTTLE --> Press **1** **ENTER**

7. Identify the location of the last sample vial.

LAST BOTTLE --> Press **3** **ENTER**

Operation

HP 3396 Integrator with an HP 5890 GC

8. Set the number of sample washes per sample vial to two.

OF SAMPLE WASHES --> Press

9. Set the number of pumps to six.

OF PUMPS --> Press

10. Set the viscosity delay to zero.

VISCOSITY --> Press

11. Set the sample size (injection volume) to 1 ml.

VOLUME --> Press

12. Set the injection speed to fast.

SLOW INJECTION --> Press

13. Set the number of solvent A washes to one.

OF SOLVENT A WASHES --> Press

14. With a tray, set the number of solvent B washes to one.

OF SOLVENT B WASHES --> Press

Without a tray, skip to the next instruction.

15. Turn the priority sample feature off.

PRIORITY SAMPLE --> Press
(1=YES)

16. Set the injection mode. CAPILLARY ON-COLUMN

Normal for capillary or packed inlet.

Press

or

On-column for an on-column inlet.

Press

Checking your work

List the parameters by pressing the keys for [LIST][SEQ].

Figure 3-8



```
* LIST: SEQ @  
  
ALS INFORMATION  
INLET SAMPLER CONTROL ..... YES  
  
EQUILIBRATION TIME IN SECONDS .... 0  
  
HP 7673C SAMPLER:  
  LOOP ADDRESS: 8  
  
ENABLE TRAY (1=YES)           1  
FRONT TOWER = INJ1  
  
FRONT INJECTOR  
  INJ/BOTTLE                   1  
  FIRST BOTTLE                 1  
  LAST BOTTLE                  3  
  # OF SAMPLE WASHES          2  
  # OF PUMPS                   6  
  VISCOSITY                    0  
  VOLUME                       1  
  SLOW INJECTION               0  
  # OF SOLVENT A WASHES       1  
  # OF SOLVENT B WASHES       1  
  PRIORITY SAMPLE (1=YES)     0  
  CAPILLARY ON-COLUMN         0  
  
*
```

HP 3396 Integrator with an HP 5890 GC.

If there are any errors, edit the parameter by pressing the keys for [EDIT][SEQ].

HP multitechnique ChemStation control with an HP 6890 Series GC

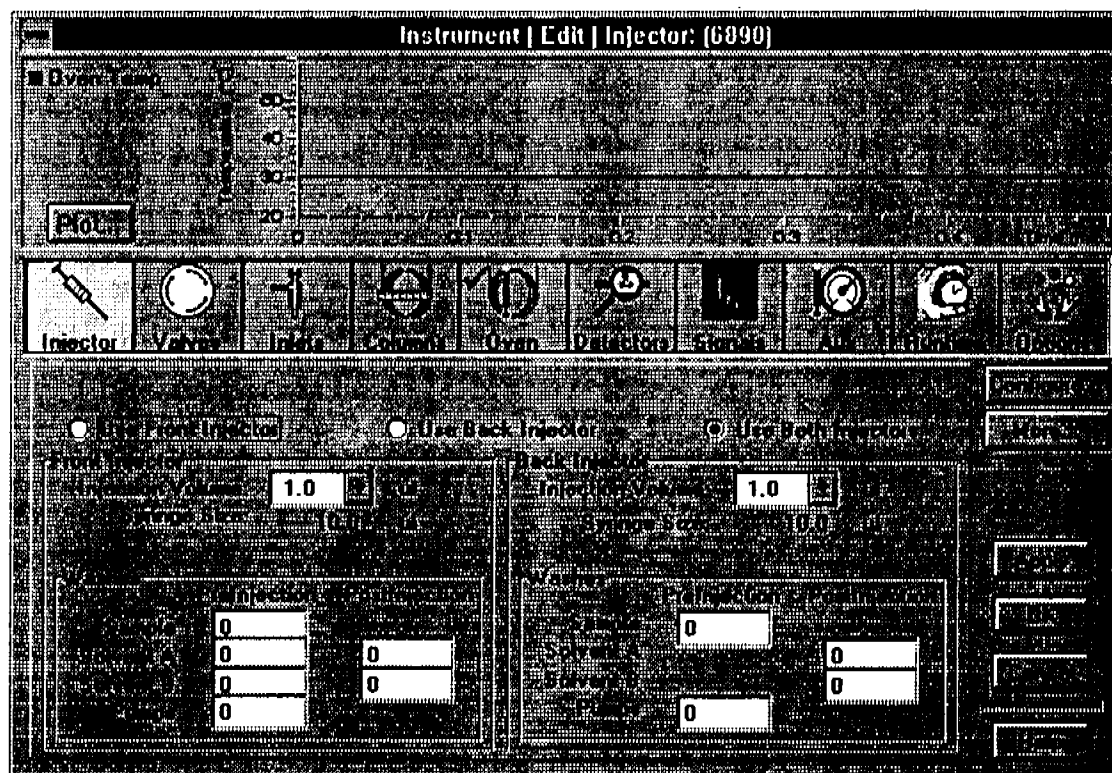
The following is an example of the HP multitechnique ChemStation control of the HP G1513A injector in the front position and an HP 18593B injector in the back position with an HP 6890 Series GC.

Injector parameters

Use the Injector Parameters dialog box to specify injector parameters for the automatic liquid sampler. To get the Injector Parameters dialog box, select Edit Parameters... from the Instrument menu.

Click on the ICON labeled Injector.

Figure 3-9



Notice the enhanced parameters are not available with the HP 18593B (back injector).

Injector parameters dialog box with an HP 6890 Series GC.

Washes

Sample (preinjection)

Number of times the syringe is rinsed with the next sample following any preinjection solvent washes. For each wash, the syringe is filled to eight-tenths its full volume (4 μl for the 5- μl syringe and 8 μl for the 10- μl syringe), and then emptied into a waste vial. Valid entries: 0 to 15. The default entry is 0.

Solvent A (pre- or post-injection)

Number of times the syringe is rinsed with solvent A. For each wash, the syringe is filled to eight-tenths its full volume (4 μl for the 5- μl syringe and 8 μl for the 10- μl syringe) and then emptied into a waste vial. Valid entries: 0 to 15. The default entry is zero.

Solvent B (pre- or post-injection)

Number of times the syringe is rinsed with solvent B. For each wash, the syringe is filled to eight-tenths its full volume (4 μl for the 5- μl syringe and 8 μl for the 10- μl syringe) and then emptied into a waste vial. Valid entries: 0 to 15. The default entry is zero.

Pumps (preinjection)

Number of times the syringe is filled with the next sample prior to injection. For each pumping stroke, the syringe is filled to eight-tenths its full volume (4 μl for the 5- μl syringe and 8 μl for the 10- μl syringe), and then emptied back into the sample vial.

Valid entries: 0 to 15. The default entry is 0.

Position

To specify the front, the back, or both injectors, click on the corresponding option button. The selection you make here determines which injector parameters (front, back, or both) become available not only at this dialog box, but also at the Extended Injector Parameters and Injector Configuration dialog boxes.

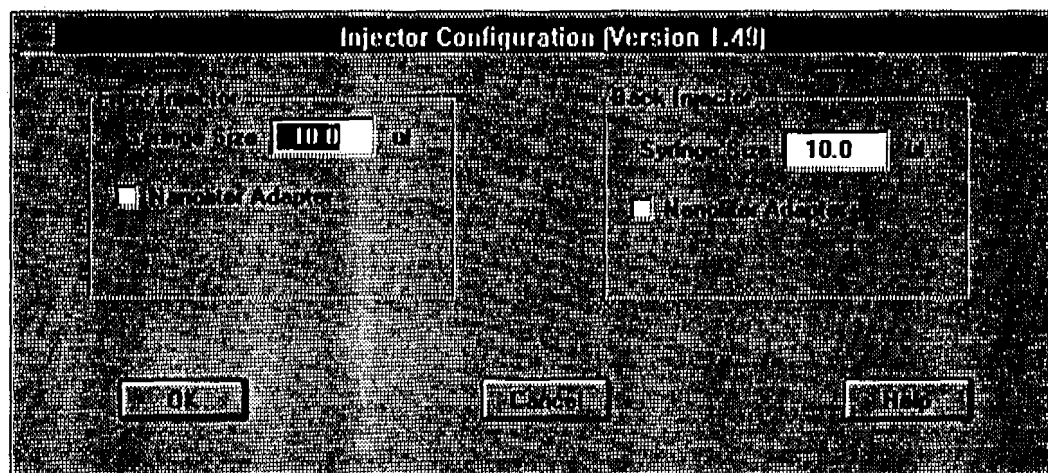
Injection Volume

Injection Volume is the volume (in μl) of sample you want to be injected. The number of stops is automatically calculated based on your entry for injection volume and syringe size. You can indicate whether or not you are using a Nanoliter Adapter at the Injector Configuration dialog box. (Click on the Configure... button.) See figure 3-10.

Syringe Size (5 μl or 10 μl)

Read-only display in this dialog box. You can modify the Syringe Size at the Injector Configuration dialog box. (Click on the Configure... button.) See figure 3-10.

Figure 3-10



Note: With the HP 5890, there is also an On-Column Injection check box.

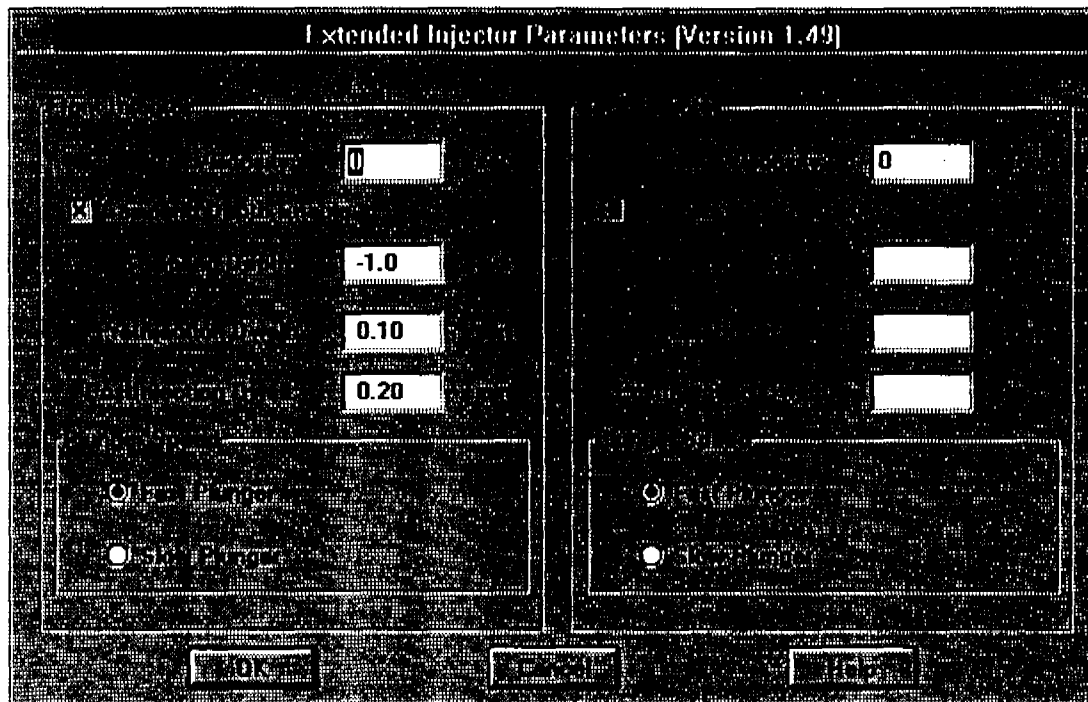
Injector Configuration dialog box.

Extended Injector Parameters dialog box

Use the Extended Injector Parameters dialog box to specify additional injector parameters for the front and/or back injectors (depending on your selection at the Injector Parameters dialog box).

Click on the [More...] button to display the Extended Parameters dialog box.

Figure 3-11



Extended Injector Parameters dialog box.

Viscosity

Number of seconds the syringe plunger should pause between the last pumping stroke and the injection stroke. For viscous samples, the pause allows the sample to flow into the vacuum that has been created in the syringe. The pause during wash strokes allows diffusion of the viscous sample into the wash solvent.

Valid entries: 0 to 7.

Sampling Depth

Depth of the needle tip in the sample vial. Click the check box next to Sample Depth Enable to enable or disable this function, then Set depth. When enabled, a sampling depth can be entered in mm as an offset from standard. (Standard equals zero.)

PreInjection Dwell

Time in hundredths of a minute for the needle to stay in the inlet before the plunger is depressed to inject sample. Valid entries: 0.00 to 1.00.

PostInjection Dwell

Time in hundredths of a minute for the needle to stay in the inlet after the plunger is depressed to inject sample. Valid entries: 0.00 to 1.00.

Plunger Speed

Speed of the syringe plunger during injection. Select a Fast Plunger or a Slow Plunger speed.

Start a Run

Open the RunControl menu, and choose Run Method (after identifying the number of the sample vial in the Sample Info dialog box). The run will not start until the chromatographic instrument is ready.

Start a sequence

Open the RunControl menu, and choose Run Sequence.

Standalone Control

Standalone Control

Standalone control means using the automatic liquid sampler's electronics to control its own operation. The controls are switches located above the START button of the injector module and are shown in figure 4-1. The HP G1512A controller has two standalone configurations, synchronous and asynchronous. (For more information on configuring the controller, see "Installing the Controller" in chapter 1.)

This chapter discusses the operation of the automatic liquid sampler with standalone control. The topics are:

- Setting the run parameters
- Running the samples
- Using two injectors
- Example of standalone control setup

The basics of operating the automatic liquid sampler are discussed in chapter 3, "Operation."

Setting the run parameters

With standalone control, use the injector control switches to set the injection parameters and the rotary switch to set the number of sample vials in the injector turret. Instructions for setting these parameters are contained in this section. For a more detailed description of each run parameter, turn to page 65.

Set the following run parameters using the switches inside the front cover of the injector. Each switch moves up and down.

- Sample size—There are two switches for 1, 2, 3, or 5 stops.
- Injection—There is one fast/slow switch.

Standalone Control

Setting the run parameters

- Number of injections per vial—There are two switches for 1, 2, 3, or 4 injections per vial.
- Number of sample washes—There are two switches for 0, 2, 6, or 10 sample washes
- On-column injection mode—There is one no/yes switch.
- Number of solvent washes—There two switches for 0, 2, 6, or 10 solvent washes.
- Position of first vial—If the tray is installed, the first vial must be in tray position 1. Without a tray, the first vial must be in turret position 1.
- Position of last vial—If the tray is installed, it starts with vial position 1 and continues until it does not find a vial in the next position. Without a tray and with more than one vial, the last vial is identified by the setting made on the rotary switch located at the top of the tower.

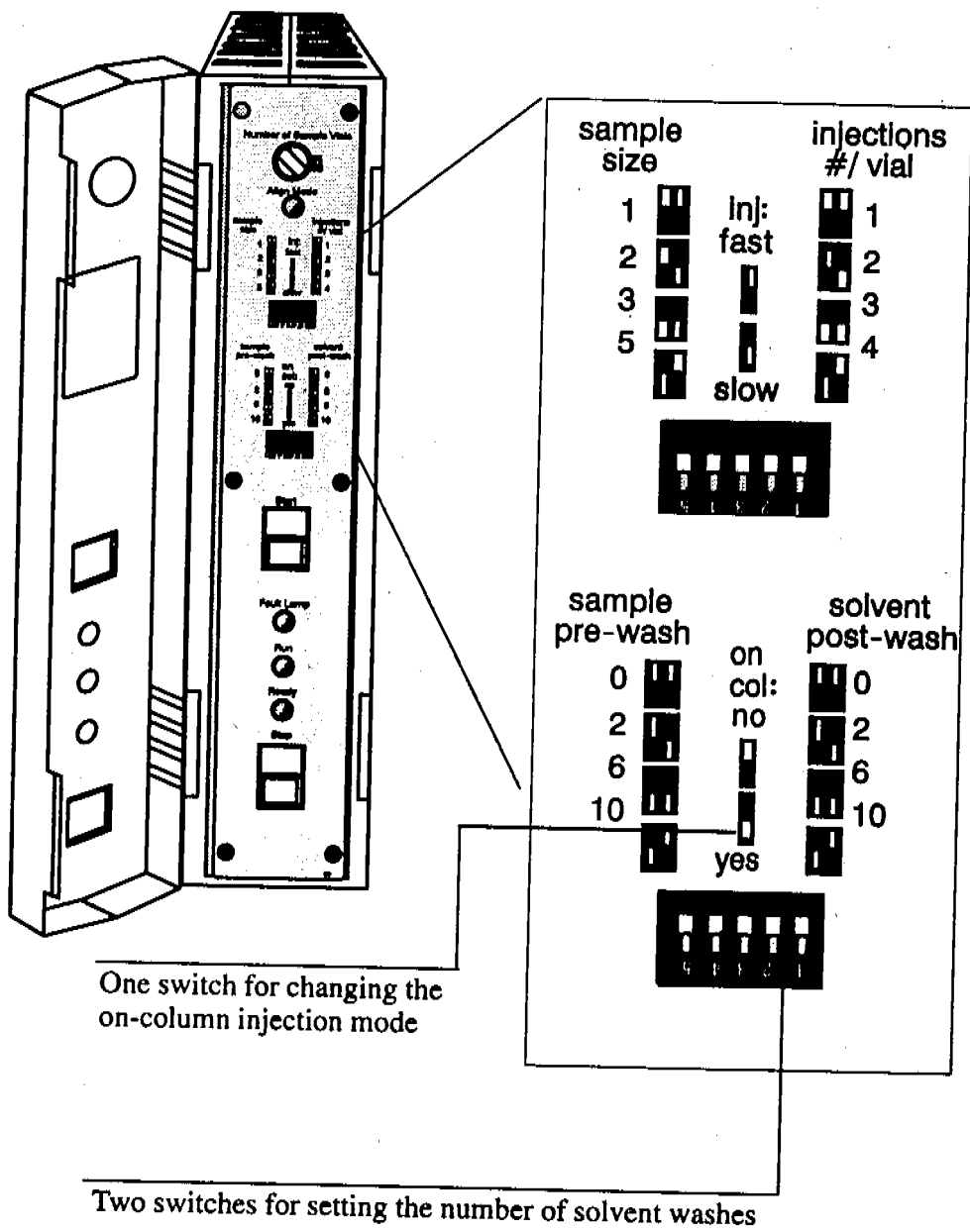
The following run parameters are either preset or not available:

- Number of pumps—This is preset to six pumps.
- Viscosity—There is 0 second delay at the top of the pump and injection strokes.
- Look for priority samples—This is not available with standalone control.
- Position—The injector position is based on the position of the injector cable connection in the controller module.

Standalone Control
Setting the run parameters

Open the door to the injector control switches (where the start and stop buttons are located). The door's hinge is on the left-hand side.

Figure 4-1







Switches for setting six Injector parameters.

Standalone Control
Setting the run parameters

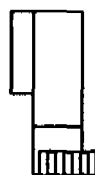
Sample size

Set the sample size with the two switches below the sample size label shown in figure 4-2. The following table shows the injection volumes for each of the switch settings.

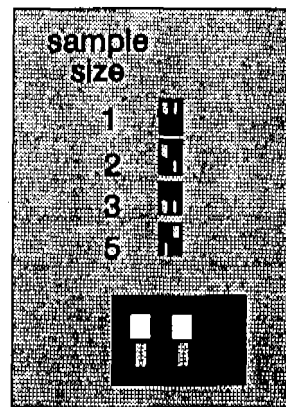
| | Switch Setting | Tenths of Syringe Volume |
|---|---------------------|--------------------------|
|  | Both up | 1 |
|  | Left up, right down | 2 |
|  | Both down | 3 |
|  | Left down, right up | 5 |

The switch settings of 1, 2, 3, and 5 correspond to tenths of a syringe volume. For example, the switch setting 2 is for an injection volume of two-tenths of the syringe volume. For a 10- μ l syringe, the volume would be 2 μ l. For a 5- μ l syringe, the volume would be 1 μ l. For a 0.5- μ l syringe, the volume is one-fifth of the syringe volume, so for switch setting 2, the volume would be 0.2 μ l.

Figure 4-2



Set for 0.5 μ l
 with a 5- μ l syringe



Switches for setting the sample size.

Caution

The injector volume for vaporizing injection ports must not produce an amount of vapor that would exceed the liner volume. Larger volumes cause sample to be lost through the purge vent and may contaminate carrier inlet lines. Refer to *GC Inlets—An Introduction*.

Standalone Control
Setting the run parameters

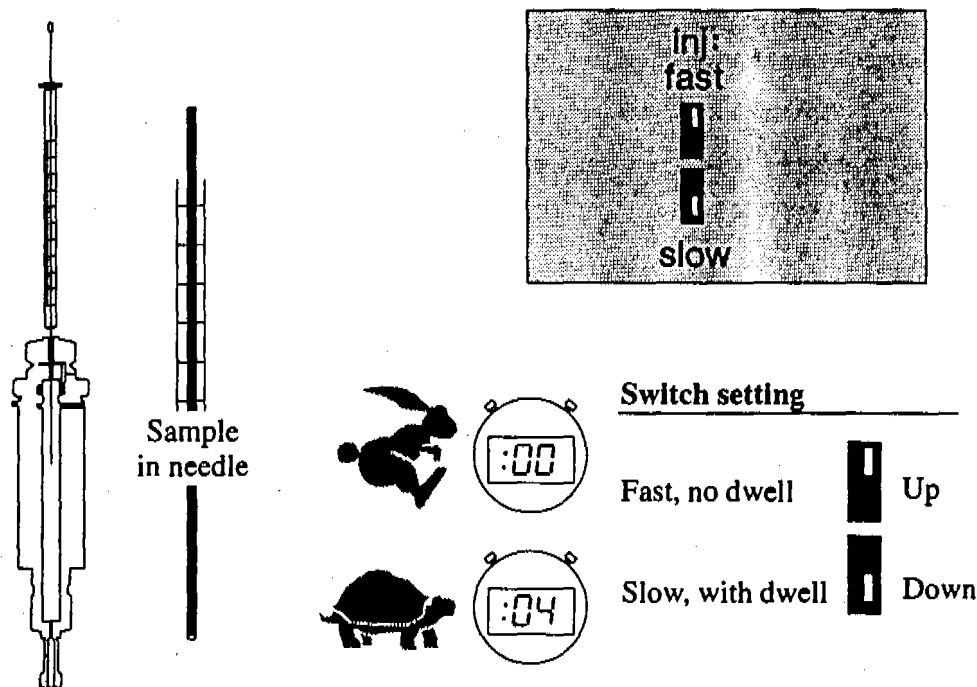
Injection

Set this switch to specify the speed of the syringe plunger during the injection strokes and whether or not the syringe needle stays in the inlet after injection of the sample. This enables you to reduce the average speed of the plunger by a factor of 18 and, with a split/splitless inlet, hold the syringe needle in the inlet for 4 seconds after the injection. This is in combination with setting the on-column mode to "no." The plunger speed during the pump and waste dispensing strokes does not change.

When you are using the on-column injection mode, this switch changes *only* the speed of the plunger (no dwell is involved).

Note: With on-column injection, the carriage speed is always slower than with other inlets.

Figure 4-3



Switches for setting the Injection speed.

Standalone Control
Setting the run parameters

Caution

The slow setting is only recommended for a few specific splitless and cool on-column applications. For most applications, set this switch to "fast." A setting of "slow" for other applications may cause peak area discrimination and reduced quantitative precision.

Number of injections per vial

Set the number of injections per vial to 1, 2, 3, or 4. The following table shows the number of injections for each of the switch settings.





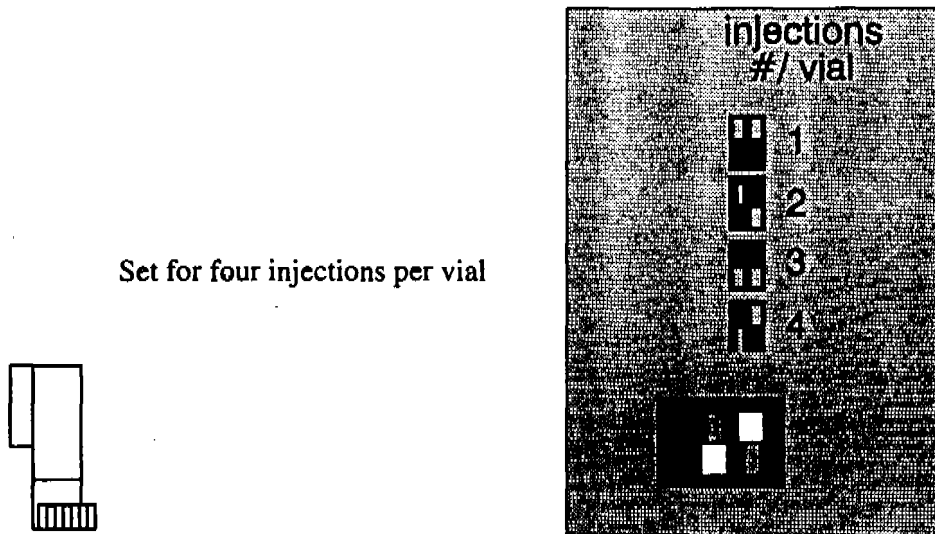
| | Switch Setting | Injections/Sample Vial |
|--|---------------------|------------------------|
|  | Both up | 1 |
|  | Left up, right down | 2 |
|  | Both down | 3 |
|  | Left down, right up | 4 |

Figure 4-4



Switches for setting the number of injections from each sample vial.

Standalone Control
Setting the run parameters

Number of sample prewashes

Set the number of sample washes to 0, 2, 6, or 10 sample washes per injection. The following table shows the number of sample washes for each of the switch settings.


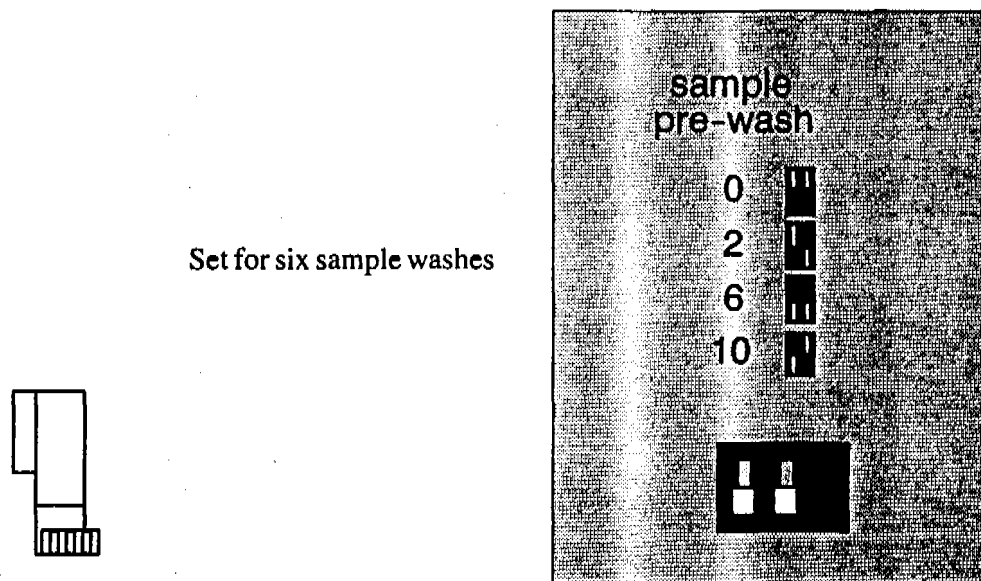
| | Switch Setting | Number of Sample Washes |
|---|---------------------|-------------------------|
|  | Both up | 0 |
| | Left up, right down | 2 |
| | Both down | 6 |
| | Left down, right up | 10 |

Figure 4-5



Switches for setting the number of sample washes.

Standalone Control
Setting the run parameters

On-column injection mode

There are two injection modes: no (normal) and yes (on-column). Normal is used with packed and split/splitless inlets. On-column mode is used with on-column inlets and changes the carriage speed and the position over the inlet.



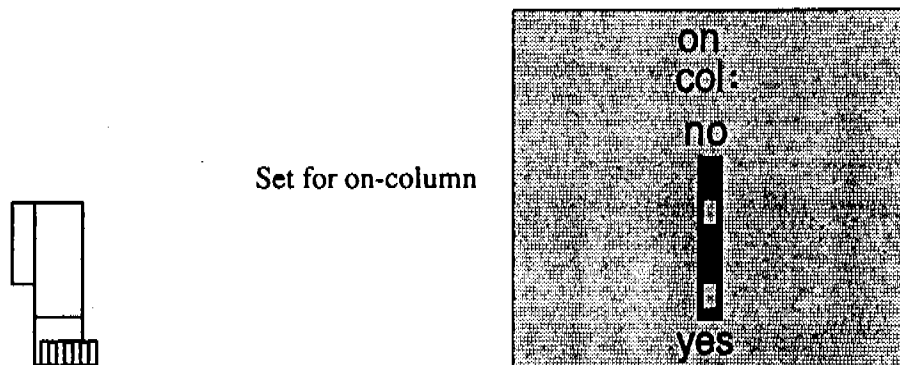
| | Switch Setting | Injection Mode |
|---|----------------|----------------------------------|
|  | Up or no | Packed and split/splitless inlet |
|  | Down or yes | On-column inlet |

Figure 4-6



Switch for setting the on-column Injection mode.

Caution

When you move the injector from one inlet to another, double-check the injection mode. Be sure it is set to the correct injection mode. If you use the wrong injection mode, you may damage the column, needle, and syringe. The chemical performance of your GC may also suffer because the carriage speed is slower.

Standalone Control
Setting the run parameters

Number of solvent postwashes

Set the number of solvent washes to 0, 2, 6, or 10 solvent washes per injection. The following table shows the number of solvent washes for each of the switch settings.

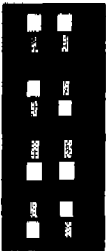
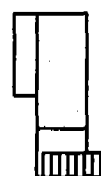
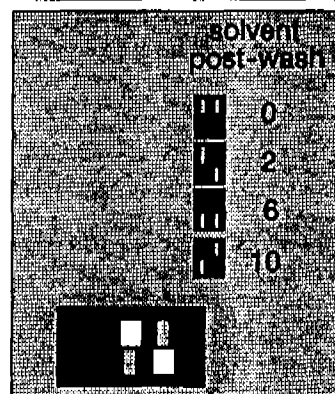
| | Switch Setting | Number of Solvent Washes |
|---|---------------------|--------------------------|
|  | Both up | 0 |
| | Left up, right down | 2 |
| | Both down | 6 |
| | Left down, right up | 10 |

Figure 4-7



Set for two solvent washes



Switches for setting the number of solvent washes.

When a tray is installed, place two solvent bottles and two waste bottles in the injector turret. The injector alternates between the solvent A and B bottles. The injector alternates dispensing waste between the two waste positions. For example, if you select two washes, the injector washes the syringe once with solvent from the solvent A bottle and once with solvent from the solvent B bottle. Without a tray, only solvent A and waste A bottles are used.

Caution

Make sure you have placed bottles in both positions. Do not exceed the injector's limit for solvent and waste volumes. If you exceed these volumes, sample carryover may affect your analysis.

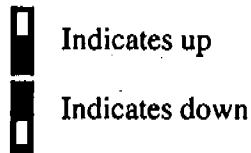
Example of setting the injector switches

Draw in the position of the switches for the following run parameters.

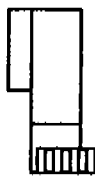
Figure 4-8

Run parameters

- 1.5 µl with a 5 µl syringe
- Fast injection
- 2 injections per sample vial
- 2 sample washes
- Split/splitless inlet
- 6 solvent washes



See page 114 for the answer.



Self-test your expertise.

| sample size | | injections #/ vial |
|--------------------------|--|--------------------|
| 1 | | 1 |
| 2 | | 2 |
| 3 | | 3 |
| 5 | | 4 |
| Inj: fast slow | | |
| | | |
| sample pre-wash | | solvent post-wash |
| 0 | | 0 |
| 2 | | 2 |
| 6 | | 6 |
| 10 | | 10 |
| on col: no yes | | |
| | | |

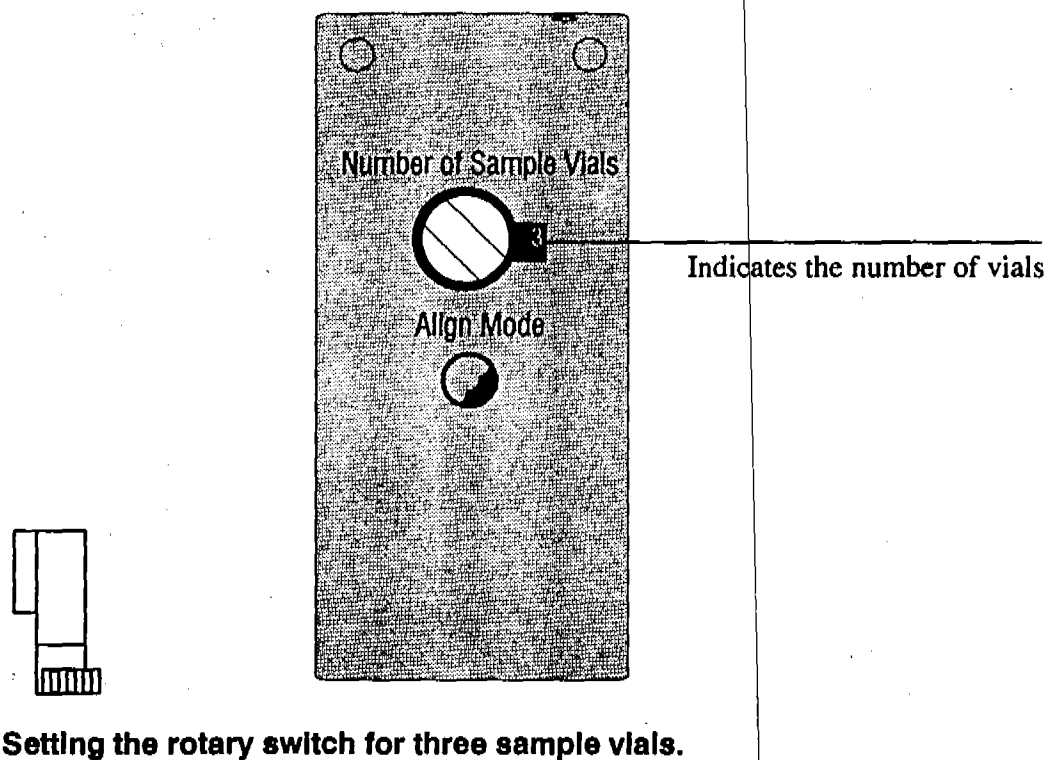
Standalone Control
Setting the run parameters

Setting the position of the last vial

When using a tray, the tray arm delivers sample vials starting from position 1 and continues to deliver vials until it cannot find the next vial in the quadrant. For example, if sample vials are in positions 1 to 10 and 15 to 30, the tray delivers samples 1 to 10 and then stops at position 11.

Without the tray, you must identify the position of the last vial in the turret with the rotary switch. Any position over three will be ignored by the standard turret. Positions 4 to 8 are only accessible when the high-density turret is mounted. The high-density turret is not compatible with the tray.

Figure 4-9



Running the samples

This section describes starting and stopping a run or sequence and the movements of the automatic liquid sampler during a run when using standalone control.

Starting a run or sequence

Start the run or sequence by pressing the start button on the injector.

What happens during a run?

From the point when you start the sequence, the automatic liquid sampler does the following:

1. The injector reads the run parameters from the switch settings and moves the turret, syringe carriage, plunger carrier, and tray to their home positions.
2. If this is the first run after turning on the power, the injector goes through the solvent wash cycle. If it is not, the injector expels any remaining sample or solvent into a waste bottle.
3. If a tray is installed, the tray picks up a vial from position 1 (or the next vial) and delivers it to the turret.
4. The injector fills the syringe with sample and dispenses it into one of the waste bottles according to the "number of sample washes" set with the control switches.
5. The injector fills the syringe with sample and dispenses it back into the sample vial six times. This is the preset number of pumps.
6. The injector fills the syringe with the "volume" of sample set with the control switches and injects it. The depth and speed of the injection stroke is determined by the injection mode switch. Just before the needle pierces the inlet septum, the injector sends a start signal to the GC.

Standalone Control
Running the samples

7. After injection, the injector washes the syringe with solvent according to the "number of solvent washes" set with the control switches.

If a tray is installed, the injector takes half of the solvent washes from the solvent A bottle and half from the solvent B bottle. The injector dispenses the waste into both waste positions. During one run, it dispenses all of the waste into one position. During the next run, it dispenses all of the waste into the other position.

If a tray is not installed, the injector takes all the washes from the solvent A bottle and expels the waste into the waste A bottle.

8. If a tray is installed, the tray returns the vial to its original position in the tray quadrant.
9. The automatic liquid sampler returns all its moving parts to their idle positions.
10. The injector waits until the GC sends a ready signal to continue.

Steps 3 through 8 are repeated according to the "injections per sample vial" set with the control switches and are repeated until the tray cannot find another vial or until the injector completes the last vial.

11. The controller gives a single beep to indicate end of sequence.

Stopping or interrupting a run or sequence

The following events interrupt a run:

- The power to the controller fails.
- You press the stop button.
- The sampler recognizes a safety or operator fault, such as:
 - You open the syringe access door.
 - The tray gripper drops a sample vial.
 - The syringe needle hits the metal side of the crimp cap.
 - The plunger binds.

With standalone control, the sampler stops immediately and aborts any sequence. If the interruption occurs after the sample is injected into the GC, the GC and the data collecting device do not complete the sample-in-progress.

If the interruption was caused by a problem that the sampler recognizes, it provides you with some feedback by a combination of flashing LED indicators. For more information, see chapter 5, "Preventive Maintenance and Troubleshooting."

The sampler does not remember which sample vial was associated with the interruption. When you restart an interrupted sequence, the sampler returns any vials that were left in the injector turrets and begins a new sequence.

To restart the sequence from the point of interruption:

1. Identify the last sample vial that was run successfully. Check the vial number of the last successful chromatogram.
2. Reposition the sample vials so that the next bottle you want to run is in position 1.
3. Press the stop button to clear any faults.
4. Press the start button.

Using two injectors

Using standalone control, you can operate two injectors and a tray. The standalone configuration of a tray with two injectors has the following characteristics:

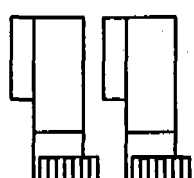
- Input and output signals are shared.
- The system sends only one INJECTOR READY signal to the GC when both injectors are ready and one START signal at the beginning of the injection stroke.
- The system sends two binary-coded decimal (BCD) signals, one for each data channel.
- The injectors operate in the synchronous mode: they inject at the same time.

Synchronous mode

In the synchronous mode, the tray arm delivers the first vial to the front injector. During the first injection, the rear injector is idle. For the second injection, the tray arm delivers the first vial to the rear injector and the second vial to the front injector.

Once both injectors have samples, they inject the samples at the same time. Figure 4-10 illustrates the sequence of injections. This mode is recommended for dual-column applications.

Figure 4-10



The diagram shows two vertical rectangular components representing injectors. Each has a series of horizontal lines at the bottom, likely representing a tray or sample holder. The two injectors are positioned side-by-side, with the front injector on the left and the rear injector on the right.

| Injection Number | Front Injector | Rear Injector |
|------------------|----------------|---------------|
| 1st | Vial 1 | Idle |
| 2nd | Vial 2 | Vial 1 |
| 3rd | Vial 3 | Vial 2 |
| nth | Vial n | Vial n-1 |
| Last | Idle | Vial n |

Synchronous N-1 Injection.

Standalone Control Using two injectors

If you want to inject the same sample into both the front and rear injectors, you must set up two vials for each sample. If you pair the vials so that 1 and 2, 3 and 4, and 5 and 6 are the same sample, the even-numbered injections correspond to injections of the same sample.

Asynchronous mode

Asynchronous standalone mode is used when there are two injectors with no tray. The injectors operate independently or asynchronously; you must press START on the appropriate injector.

The APG remote is redefined to provide an independent Start, Ready in, Ready out, and Start Request signals for each injector. To configure your APG remote connector for asynchronous operation, see figure 4-11.

Figure 4-11



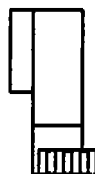
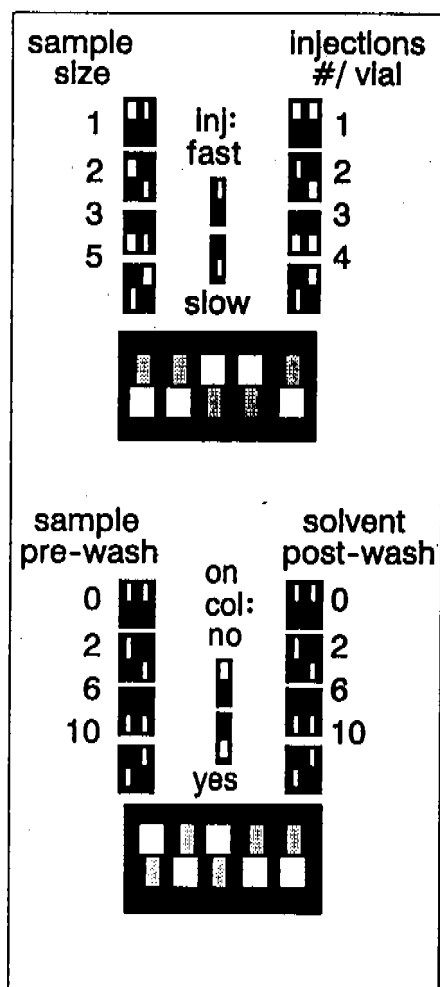
| APG Remote Pin Out | General-Purpose Cable |
|---------------------------|-----------------------|
| Pin 1—Ground | Black |
| Pin 2—Front Ready In | White |
| Pin 3—Back Ready In | Red |
| Pin 4—Front Ready Out | Green |
| Pin 5—Back Ready Out | Brown |
| Pin 6—Front Start Request | Blue |
| Pin 7—Back Start Request | Orange |
| Pin 8—Front Start Out | Yellow |
| Pin 9—Back Start Out | Violet |

General-purpose cable (HP part no. 35900-60670).

The asynchronous standalone control configuration with two injectors but without a tray is only supported by Hewlett-Packard for isothermal analyses (see figure 4-12). Because there is no direct connection to the GC, this system only waits for readiness from an analog-to-digital (A/D) converter or other data handling device.

Standalone Control
Using two Injectors

Figure 4-12



Answers to the self-test.

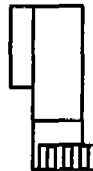
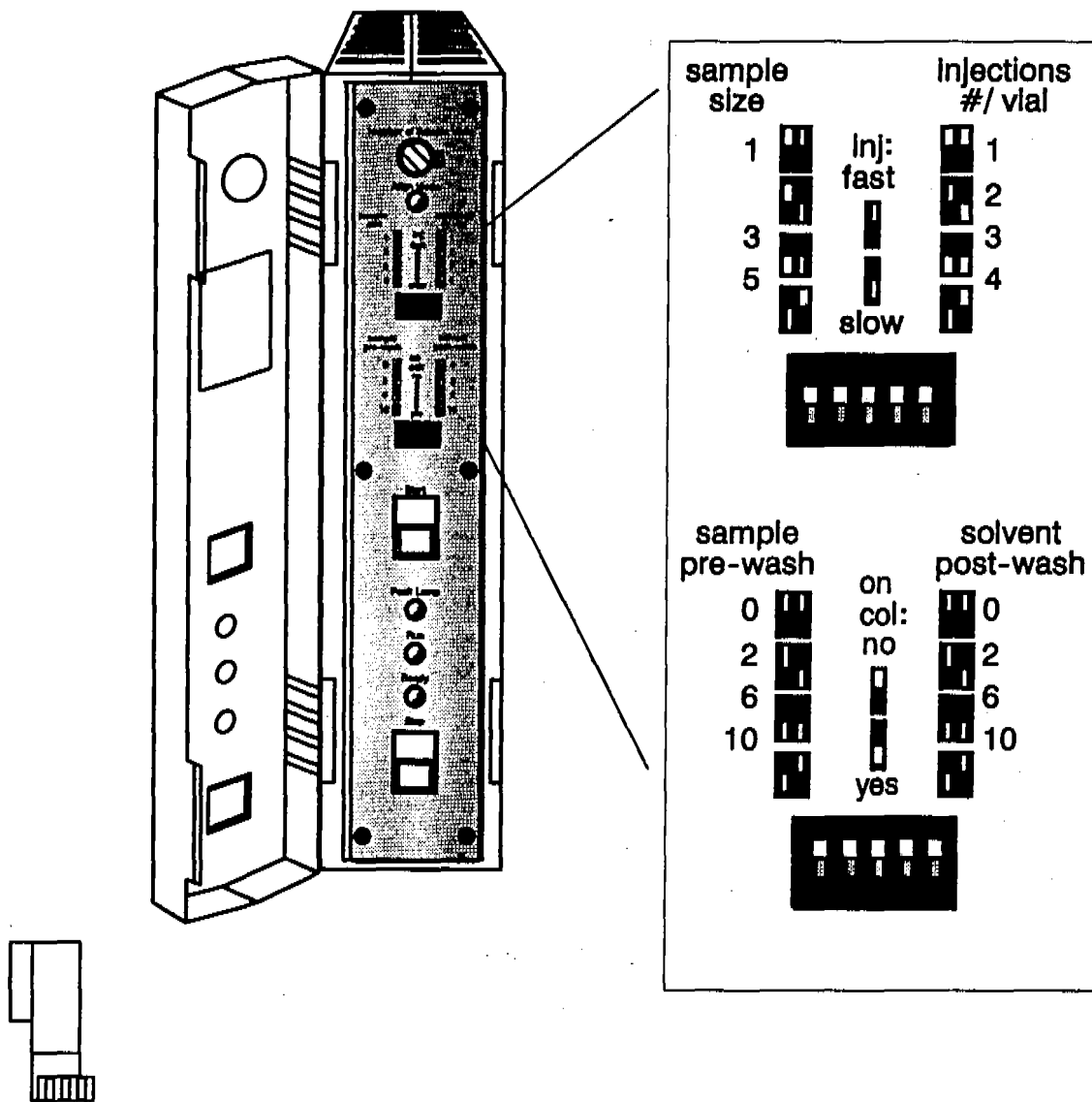
Switch settings for the example on page 107:

- 1.5 μ l with a 5- μ l syringe
- 2 injections per sample vial
- Injection set to "fast"
- 2 sample washes
- 6 solvent washes
- On-column injection mode set to "no"

Example of standalone control setup

1. Set the GC to minimize the time between injections.
2. Open the door to the injector control switches.

Figure 4-13



Switches for setting six injector parameters.

Standalone Control

Example of standalone control setup

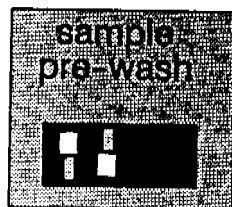
3. Set the number of injections per sample vial to one.

Both switches up.



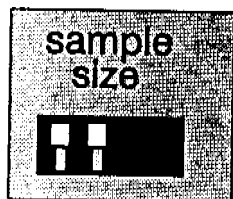
4. Set the number of sample washes per sample vial to two.

Left switch up,
right switch down.



5. The number of pumps is preset to six.
6. The viscosity delay is preset to zero.
7. Set the sample size (injection volume) to 1 μ l.

Both switches up.



8. Set the number of solvent A washes to two.
 - a. With a tray, the two solvent washes are split so that the injector performs one solvent A and one solvent B wash.
 - b. Without a tray, the injector performs two solvent A washes.

Standalone Control

Example of standalone control setup

9. Set the injection mode

For a packed or split/splitless inlet, switch up.

For an on-column inlet, switch down.

on
col:

no



yes

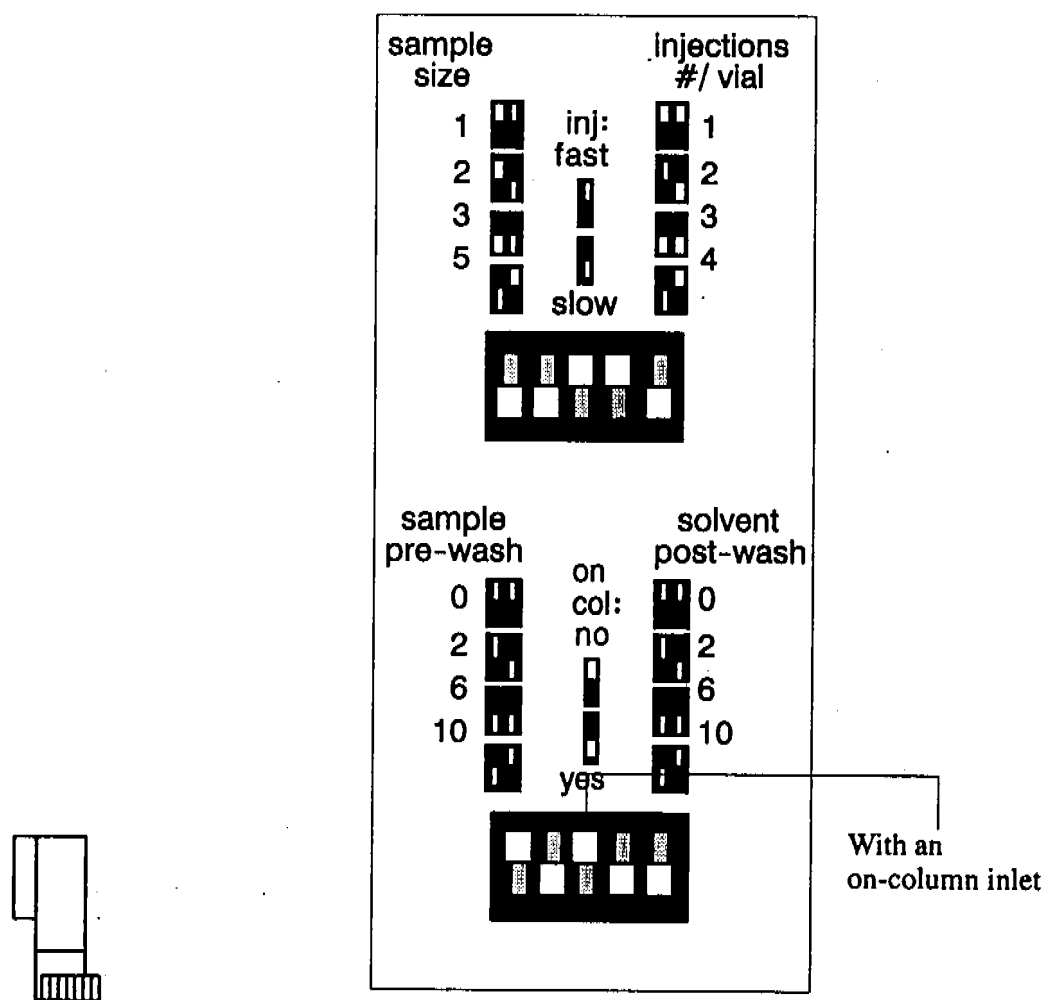
Checking your work

Before you start the sequence, check the run parameters.

Standalone control

Check the injector control switches that you set. The switches should look like the following:

Figure 4-14



Example of switch setting.

If there are any errors, change the appropriate switches.

Preventive
Maintenance and
Troubleshooting

Preventive Maintenance and Troubleshooting

Preventive maintenance

This section contains some suggestions for ensuring good performance. The maintenance interval varies with the use of the instrument.

Caution

Do not use any lubricants on the automatic liquid sampler. They may affect the chemical performance of the GC and damage the instruments.

For instructions on the proper use and replacement of consumable parts, such as septa, inlet liners, syringes, and sample vials, see chapter 3, "Operation." For instructions on aligning the brackets, see the bracket installation guides.

Regular basis (before you start a sequence)

- Get a new or clean syringe rinsed with solvent.
- Be sure the plunger carrier holds the plunger button.
- Measure 4.5 ml of fresh solvent in each solvent bottle.
- Be sure the waste bottles are clean and empty.
- Be sure the number of sample vials does not exceed solvent capacity.
- Place the sample vials correctly according to the controlling device.
- Cap the sample vials properly.

Note: The inlet does not require maintenance.

Occasional basis

- Clean the surface of the tray arm, gripper, gripper jaws, and tray quadrants.
- Vacuum off any dust that builds up around the vents on the front and back of the controller.
- Clean the needle guide and nearby surfaces of the injector. Dust and dirt accumulate in these areas and can be picked up easily by the syringe needle and carried into the inlet.
- Clean the sample vial inserts for the turret.
- Clean the surface of the injector. Vacuum off any dust that builds up around the vents of the electronics assembly.
- Realign and tighten the screw, standoffs, and mounting posts of the injector bracket.
- Tighten the screws and standoffs of the tray bracket.
- Be sure all the cables are attached securely to the back of the power supply.
- Be sure the tray is positioned over the alignment pin and locked in place.

Turret exchange

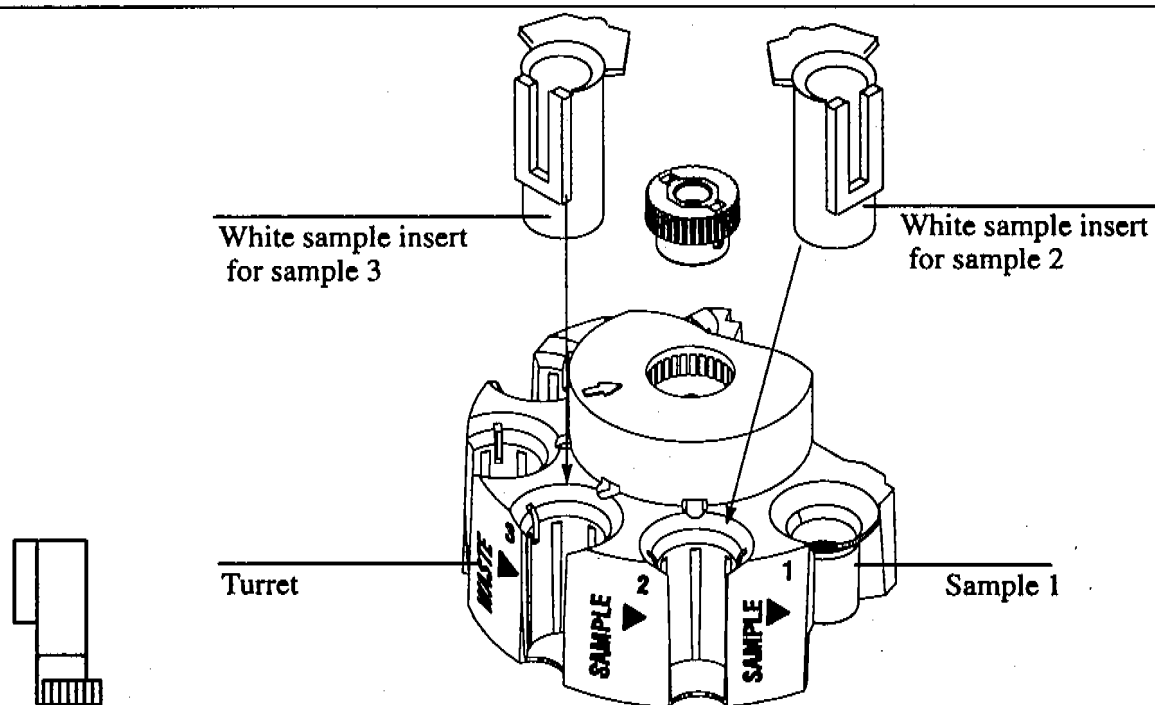
If you need to remove or exchange your turret, use the following instructions for proper replacement.

1. Turn off the controller if the injector is installed.
2. Open the tower door. Unscrew and remove the knurled nut from the top of the turret. (See figure 5-1).
3. Rotate the turret so that the open section faces the back of the tower and the arrow on top of the turret points directly back. (See figure 5-1.) Push the stripper arm toward the back, lift the turret up so that it clears the center shaft, and remove it from the front of the tower.
4. To install the replacement turret, align the pin on the motor shaft with the slot located on the underside of the turret.

Preventive Maintenance and Troubleshooting
Preventive maintenance

5. Push the stripper arm to the back of the tower, insert the turret with the open section facing the back of the tower with the arrow pointing back, and install the turret with the pin seated in the slot. Seat the turret on the shaft, rotating it gently, if necessary, until it drops completely into position. The top of the threaded shaft should be almost level with the top of the turret, and the stripper arm should clear the top by approximately 1 mm as the turret is rotated.
6. Replace the knurled nut, and tighten it firmly (finger-tight).
7. Restore power, and check for the green tower ready light. For proper needle depth in the sample vial, the syringe carriage height calibration must be carried out each time the turret is changed. See the following section, "Alignment procedure."

Figure 5-1



Placing sample inserts for three sample vials.

Alignment procedure

After changing a turret, it is recommended to perform the alignment procedure. If the yellow Align LED is on the injector will not operate until this procedure has been performed. Use the following instructions to perform the alignment procedure.

1. Turn the controller off. Put the injector into diagnostic mode by simultaneously pressing the START and STOP buttons on the front of the injector while turning the controller on. Hold the START and STOP buttons down until the LEDs come on. They will be lighting in rotating pattern.
2. Remove the syringe, and carefully remove the needle support rod from the injector.
3. Press the STOP button. The red LED will start flashing a double flash. Press STOP three times more, moving through displays of three, four, and five flashes. The sequence of five flashes is the starting point for alignment.
4. Press START to begin the alignment procedure. The injector will go through the following three steps:
 - The turret homes (this is a clockwise and then a counterclockwise continuous motion).
 - The syringe carriage starts moving up and down repeatedly to set a position relative to the inlet.
 - The syringe carriage is stepped down until it touches the turret. This sets the position relative to the turret.

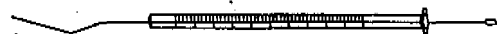
When the alignment is complete, the red LED will return to two flashes, and the yellow Align LED will go out. Power-cycle the controller to return it to the ready state. Return your syringe and needle support rod to their original positions.

Troubleshooting

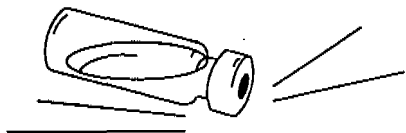
Find the illustration in figure 5-2 that best identifies your problem, and then turn to the appropriate page.

For information on how to obtain service from Hewlett-Packard, see page 153.

Figure 5-2



Bent syringe needle 125



Dropped sample vial 126



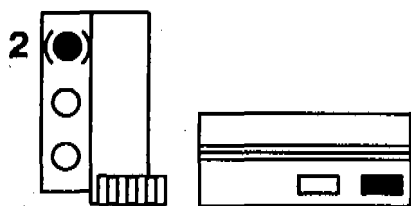
Variability 127

Contamination or ghost peaks 129

Peak area discrimination 132

Sample carryover 134

No signal/no peaks 135



Fault light symptoms 136

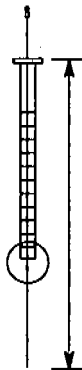
REAR INJECTOR INCOMPLETE INJECTION Error messages 151

Troubleshooting map.

Bent syringe needle



When you find a bent syringe needle, check:

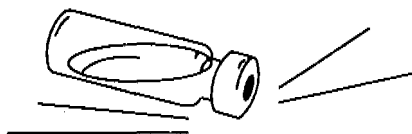


- Was the syringe installed properly in the syringe carriage?
- Was the syringe needle straight? Roll the syringe on a flat surface, and watch the tip of the needle.
- Are you using the correct syringe? Is the combined length of the syringe barrel and needle about 126.5 mm? For more information, turn to “Selecting and installing syringes” on page 53.
- Does the injection mode parameter match the type of inlet under the injector module—“normal” for packed or split/splitless inlets, “on-column” for on-column inlets? For more information, see “Injection mode” on page 69.
- Are the needle support and needle guide cone clean? Remove any residue or septum deposits. For more information, see “Preventive maintenance” on page 120.
- Is the GCs septum nut too tight? For more information, see “Changing septa” on page 59.
- Is the septum of the crimp cap centered over the sample vial? For more information, see “Crimp capping sample vials” on page 35.
- Are the inside diameters of the sample vial, microvial insert, and vial cap septum at least 5 mm? For more information, see “Specifications” in chapter 2.

WARNING

When troubleshooting the injector, keep your hands away from the syringe needle. The needle is sharp and may contain hazardous chemicals.

Dropped sample vial



When you find a mishandled sample vial, check:

- Are there folds or wrinkles in the crimp cap especially near the neck of the sample vial? For more information, see “Crimp capping sample vials” on page 35.
- Are you using the correct sample vials? Is the distance from the vial bottom to the top of the neck about 28.4 mm? For more information, see “Specifications” on page 32.
- If you use labels on the sample vials, are they the correct size? For more information, see “Selecting and labeling sample vials” on page 31.
- The installation of the tray bracket on the injector bracket. Specifically, check the location of the three standoffs used to attach the tray bracket to the injector bracket. See the installation guide for the tray bracket.
- The installation of the tray on the mounting bracket. Be sure that it is pushed all the way to the alignment pin and locked in place. See “Mounting the tray” on page 18.
- For obstacles to the tray arm or injector turret. Remove any obstacles.
- The condition of the tray quadrants and turret. Clean any residue from the sample positions. If a temperature-controlled liquid is being pumped through the quadrants, see “Controlling sample vial temperatures” on page 174.

Chromatographic symptoms

You may suspect a problem in the automatic liquid sampler based on what you see in a chromatogram or by comparing chromatograms. Each of the five parts of this section is named after a chromatographic symptom. Each part contains a list of possible causes and recommended solutions.

It is often difficult to identify a cause. For this reason, the causes are listed from most common to least common.

If you cannot correct the problem, obtain HP service.

Variability

Figure 5-3



Retention times or areas are not reproducible.

Inlet septum is leaking

If the septum is leaking, replace the septum. If the leaking septum has experienced less than 200 injections, check for the following:

- The septum retainer nut is too tight. See "Changing septa" on page 59.

Preventive Maintenance and Troubleshooting

Chromatographic symptoms

- The syringe needle is not straight. See “Inspecting syringes” on page 56.
- The syringe is not installed correctly. See “Installing syringes” on page 57.
- The injector bracket is not aligned to the injection port. See the appropriate link manual that describes the installation.

Syringe is worn or dirty

If the syringe looks dirty or the plunger is sticking, clean the syringe with an appropriate solvent, or follow the syringe manufacturer’s cleaning instructions.

Sample volume is too low or too high

If the sample vials are not filled correctly, evaporation or contamination may affect the analysis. The sample level should be approximately half of the volume of the vial. See “Filling sample vials” on page 34.

Vial caps are loose

If you can turn the vial crimp caps by hand, they are too loose. Loose caps may cause volatile samples to change concentrations over time. See “Capping sample vials” on page 35.

Sample is not stable

Some samples change with heat or ultraviolet light. Check the sample’s stability. There are several ways to reduce changes to unstable samples:

- Use the injector fan to cool the sample waiting in the injector turret.
- Use the tray quadrants to cool the sample.
- Use amber sample vials.
- Store the samples in a protected environment.

Sample size varies

If the sample size varies, the syringe is probably not precise or the plunger is worn. Variations may be due to syringes with removable type needles because of dead volumes or needle-to-needle variation.

Air bubbles are in the needle

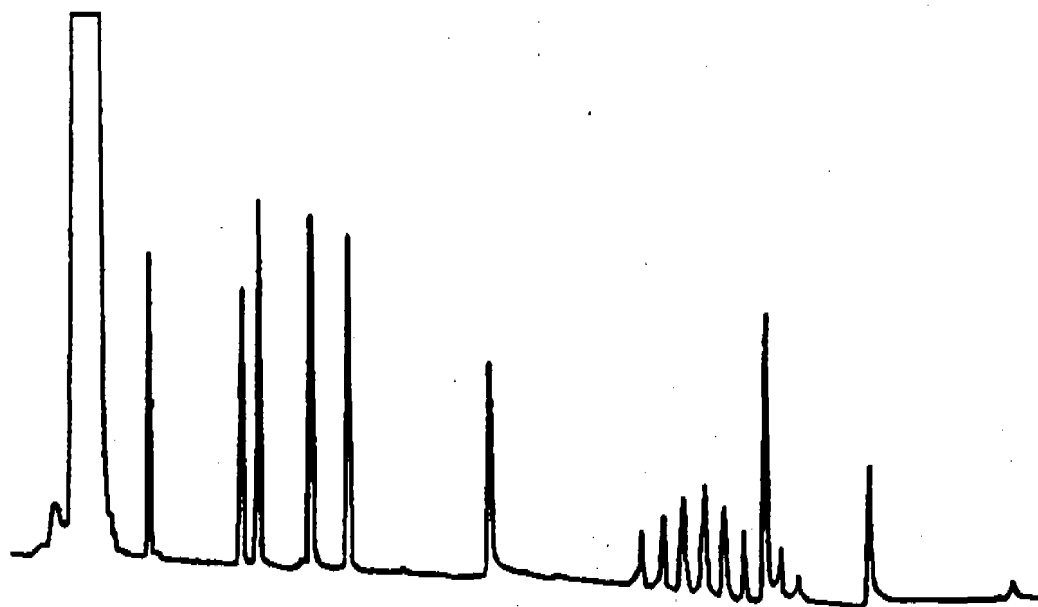
If air bubbles are in the needle, increase the run parameter that controls the number of pumps. See "Number of sample pumps" on page 66.

If this does not help and the sample is viscous, try the following:

- Reset the viscosity run parameter. See "Viscosity delay" on page 66.
- Use the tray quadrants to warm the sample.
- Dilute the sample in the appropriate low-viscosity solvent.

Contamination or ghost peaks

Figure 5-4



Contamination or ghost peaks.

Vial cap septum is dissolving in solvent

Ghost peaks sometimes appear in the chromatogram when small pieces of vial septum material dissolve in the sample. Make several "blank" runs to determine the presence or absence of the ghost peaks. Check for the following:

- If the vial septum is not flat, the needle tends to core the septum and drop pieces into the sample. See "Capping sample vials" on page 35.
- If the syringe needle has burrs, it could cut pieces of the septum and push them into the sample. See "Inspecting syringes" on page 56.
- If the vial septum is not resistant enough to the solvent you are using, try a more resistant type.

Sample vials are contaminated

Ghost peaks are sometimes caused by contaminated sample vials. Try new or clean vials to see if ghost peaks disappear. Store new vials in a contamination-free location.

Injection port septum is giving off volatiles

Make several "blank" runs with a small piece of aluminum foil backing the inlet septum. If the contamination peaks disappear, they were probably due to the septum. Try replacing the septum you usually use with another type.

Column is contaminated

High molecular weight samples that contain residues may cause the syringe, the inlet liner, or the first few inches of column to become contaminated. Do the following:

- Replace or clean and deactivate the inlet liner.
- Examine the first few inches of a capillary column for foreign material by holding a light behind it. If possible, remove the contaminated section.

Preventive Maintenance and Troubleshooting
Chromatographic symptoms

Sample is not stable

Some samples change with heat or ultraviolet light. Check the sample's stability. There are several ways to reduce the change:

- Use the tray quadrants to cool the sample.
- Use amber sample vials.
- Store the samples in a protected environment.

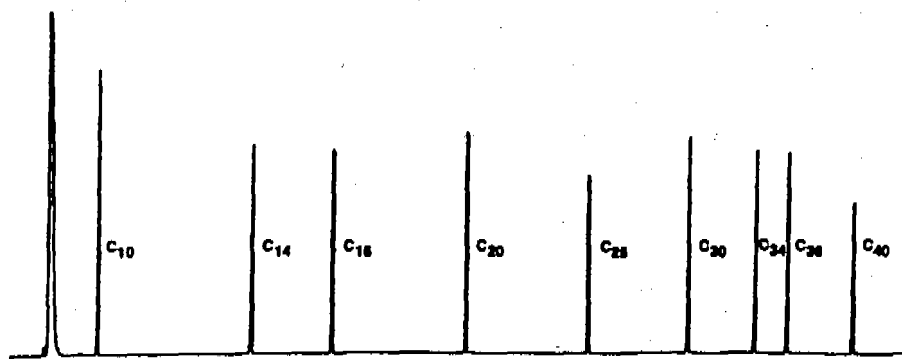
Impurities in the carrier gas

Install a carrier gas trap in the gas line to the GC, or replace the existing trap. If the gas contains impurities, the contamination peaks gradually disappear once the trap is installed.

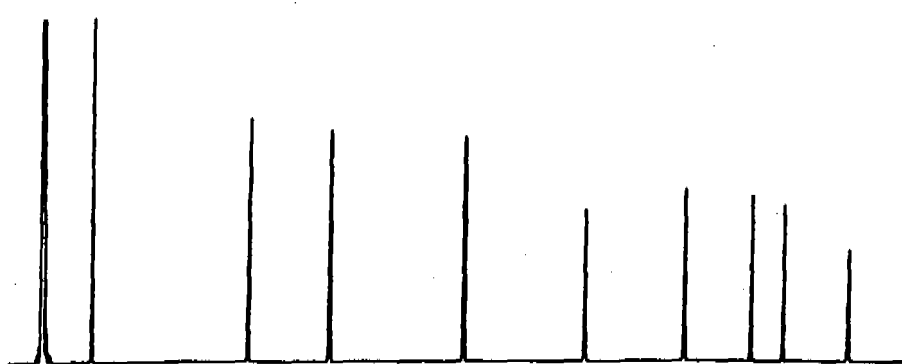
Peak area discrimination (smaller or larger peaks than expected)

Figure 5-5

Automated
injection



Manual
Injection



Smaller or larger peaks than expected.

You are comparing a chromatogram without needle fractionation against one with needle fractionation

In the normal injection mode, the automatic liquid sampler uses fast injection to deliver a representative amount of the sample. Fast injection minimizes needle fractionation. Without fast injection, sample boils out of the needle. Chromatograms from manual injection or slower auto injection devices show higher levels of low molecular weight versus higher molecular materials because the low boilers boil out of the needle faster than the high boilers.

Preventive Maintenance and Troubleshooting
Chromatographic symptoms

You are using a packed inlet and a 530- μ m column

Capillary columns used with packed inlets do have some inherent sample discrimination characteristics. See "Suggestions for packed inlets with 530- μ m columns" on page 60.

You are using a packed column

Double-check the following:

- Is the inlet temperature correct for this particular sample?
- Is the sample reacting with the inlet liner or fittings?

There is a leak in the GC system

Replace the septum, and check the fittings for leaks. If the leaking septum has experienced less than 200 injections, check for the following:

- The septum retainer nut is too tight. See "Changing septa" on page 59.
- The syringe needle is not straight. See "Inspecting syringes" on page 56.
- The syringe is not installed correctly. See "Installing syringes" on page 57.
- The injector bracket is not aligned to the injection port. See the appropriate link manual that describes the installation.

Sample is not stable

Some samples change with heat or ultraviolet light. Check the sample's stability. There are several ways to reduce the change:

- Use the tray quadrants to cool the sample.
- Use amber sample vials.
- Store the samples in a protected environment.

Preventive Maintenance and Troubleshooting
Chromatographic symptoms

Vial caps are loose

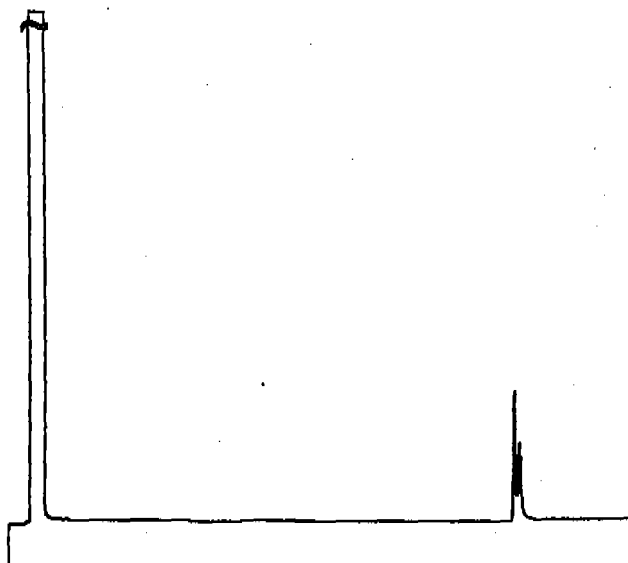
Loose vial caps can cause selective loss of lighter materials from a sample. The caps should not rotate easily if installed properly. See "Capping sample vials" on page 35.

Detector response has changed

Recalibrate the detector. Absolute response factors can change several percentage points during the day depending on the type of detector in use.

Sample carryover

Figure 5-6



Blank run showing carryover peaks.

Number or type of washes is insufficient

Check the run parameters for the number of sample and solvent washes. The number of washes you need depends on your application. See "Controlling sample carryover" on page 51.

You ran out of solvent

Check the solvent bottles. If the solvent level is below 2.5 ml, the syringe cannot reach the solvent. Replace the remaining solvent with 4 to 4.5 ml of fresh solvent. See "Filling and placing the bottles" on page 41.

Check the waste bottles. If the waste level is near the neck of the bottle, replace it with an empty bottle.

Syringe is worn or dirty

If the syringe looks dirty or the plunger is sticking, clean the syringe with an appropriate solvent, or follow the syringe manufacturer's cleaning instructions. If the syringe seems worn, replace it.

Samples (vial-to-vial) are of immiscible types

In this situation, the sample and solvent washes may not rinse the syringe properly. Either increase the number of wash cycles or use a solvent that rinses a variety of sample types.

No signal/no peaks

Syringe plunger is malfunctioning

Verify that the button of the syringe plunger is fastened properly by the plunger screw. If the plunger screw is loose, tighten it. See "Installing syringes" on page 57.

Also, check the syringe needle for plugging. If the syringe is plugged, replace or clean the syringe.

Sample level is too low in vial

If there is no or very little sample in the vial, the needle may not be able to reach it. See "Filling sample vials" on page 34.

Sample is viscous

If the sample is viscous, try the following:

- Increase the viscosity run parameter. See "Viscosity delay" on page 66.
- Use the tray quadrants to warm the sample.
- Dilute the sample in the appropriate low-viscosity solvent.
- Turn off tower fan.

Fault light symptoms

Four light-emitting diodes (LEDs) on the injector indicate the status of the injector. Two LEDs on the controller module indicate the status of the controller and the tray if it is installed.

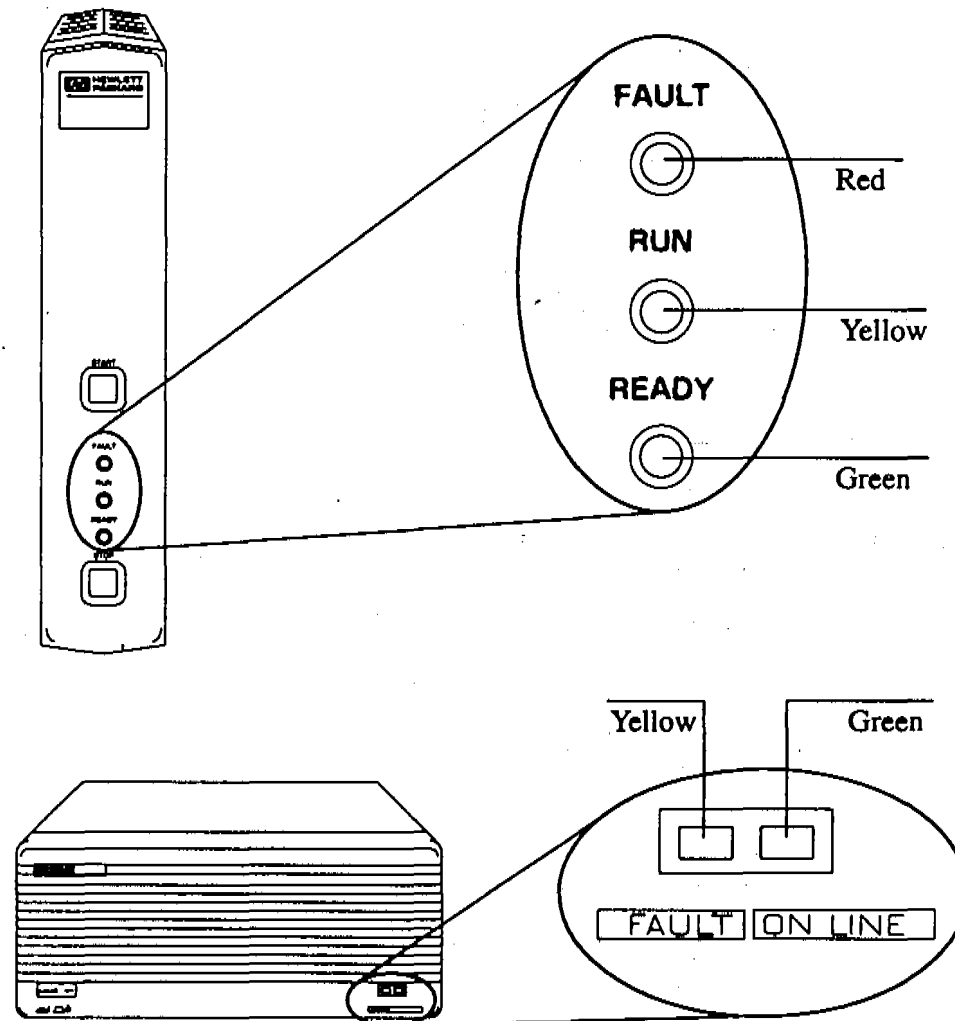
During normal operation, the controller's green LED and the injector's green LED are on. If the injector is busy, the injector's yellow LED is also on.

When a problem occurs on the injector, the red LED flashes. When a problem occurs with the turret alignment, the yellow Align LED is on (located behind the injector front panel). When a problem occurs on the tray or controller, the yellow LED on the controller flashes, and a beeper sounds in synchronization with the flashes.

If the automatic liquid sampler is controlled by an external device (i.e., not standalone), the controller sends error messages to the controlling device. For example, the ChemStation software displays an error message. The controller LEDs do not flash.

Fault light locations, colors, and patterns

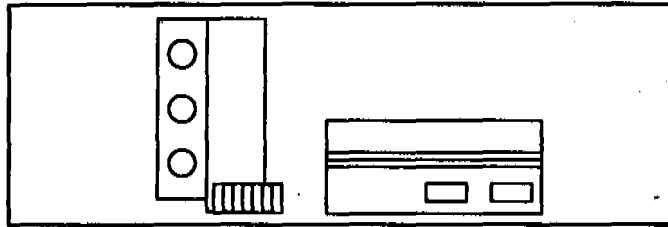
Figure 5-7



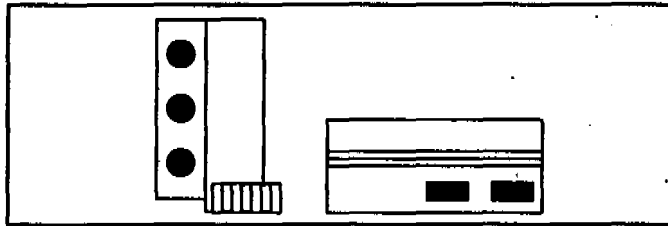
Location and pattern of LEDs during normal operation.

Preventive Maintenance and Troubleshooting
Fault light symptoms

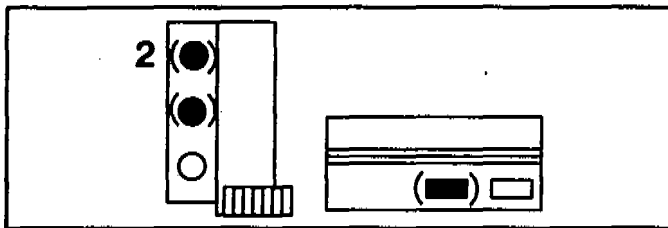
Throughout the rest of this section, an icon is used to represent the pattern of the fault lights and beeps. The following are examples of the four basic patterns. A number indicates the number of flashes or double flashes that occur between the pauses. For example, the 2 indicates that the red injector LED flashes twice, then pauses, then flashes twice, and repeats this pattern until the fault is cleared or the controller is turned off.



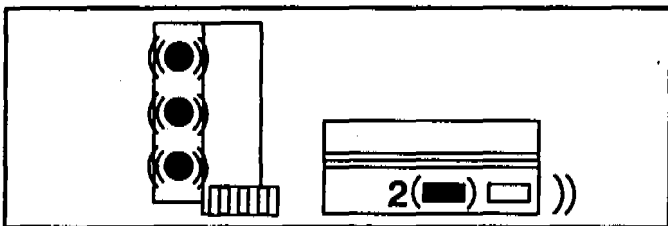
LEDs off



LEDs on



Red and yellow LED blinking



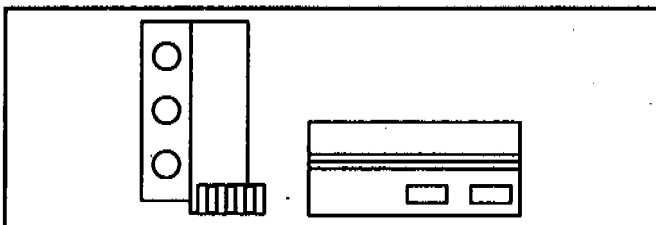
LEDs double-blinking and beeping

Responding to the fault lights

Use the following instructions to try to solve the problem before obtaining HP service:

1. Find the icon on pages 139 to 150 that describes the LED pattern on the injector and controller.
2. Read the information under the icon. Try the suggested actions.
3. If you cannot find the pattern or the suggested actions do not correct the problem, obtain HP service. See page 153.

Power error



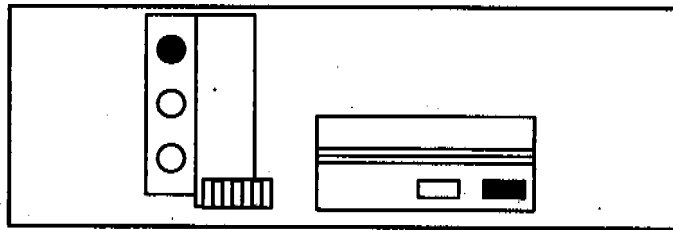
Probable causes

- The line voltage is off.
- The +5 V supply on the main controller board is not operating.

Suggested actions

1. Check the power source for your bench.
2. Check to see that the power cord is installed and the power switch is on.
3. Obtain HP service.

Injector door open/not mounted



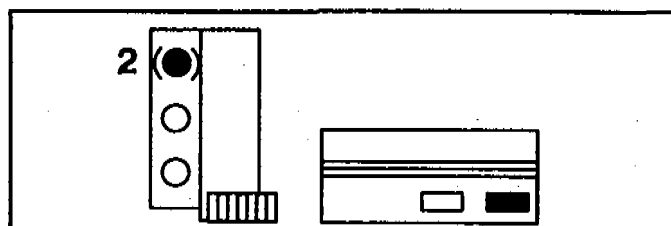
Probable causes

- The injector door is open.
- The injector is mounted incorrectly on the bracket.

Suggested actions

1. Ensure that the injector door is closed.
2. Ensure that the injector is mounted properly. For more information, see "Installing the injectors" on page 11.
3. If the fault light stays on, obtain HP service.

Syringe Error



Probable causes

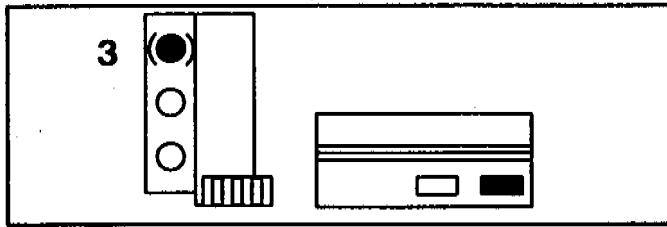
- The syringe carriage motor has a problem.
- The syringe carriage cannot find its home position.
- The syringe carriage sensor is inoperable.
- The improper injection mode was used.

For information on the syringe carriage home positions, see the "Glossary."

Suggested actions

1. Ensure the syringe is installed correctly. For more information, see "Selecting and installing syringes" on page 53.
2. Ensure the syringe meets specifications.
3. Check the on-column injection mode parameter. If the parameter was set incorrectly for on-column injection, check for damage to the inlet liner and column.
3. If the syringe needle is bent, also check the list of actions on page 125.
4. Press the STOP button on the injector to clear the fault indicators. Restart the sequence.
5. If the error occurs again, obtain HP service.

Turret error



Probable causes

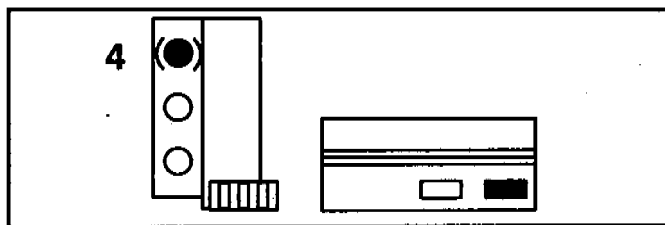
- Something has interfered with the turret rotation.
- The turret motor/encoder assembly is inoperable.
- The turret type was changed without performing the alignment procedure on the injector.

For information on the turret's home positions, see the "Glossary."

Suggested actions

1. Clear any obstructions from the turret.
2. Press the STOP button on the injector to clear the fault indicators. Restart the sequence.
3. Check the Align LED. If it is lit, perform the alignment procedure. (See the "Alignment procedure" in this section.)
4. If the error occurs again, obtain HP service.

Plunger error



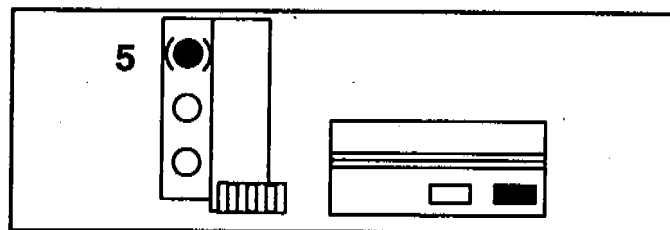
Probable causes

- The plunger carrier is out of position.
- The plunger carrier sensor(s) is inoperable.
- The plunger motor wires are disconnected.
- The plunger carrier is operating incorrectly.
- The plunger solenoid is binding.

Suggested actions

1. Remove the syringe, and check it for plunger stickiness or binding. Replace the syringe if necessary. For more information, see "Selecting and installing syringes" on page 53.
2. Check the viscosity of the sample against the viscosity parameter. Reset the viscosity parameter if necessary.
3. Press the STOP button on the injector to clear the fault indicators. Restart the sequence.
4. Connect the wires.
5. If the error occurs again, obtain HP service.

Incomplete injection



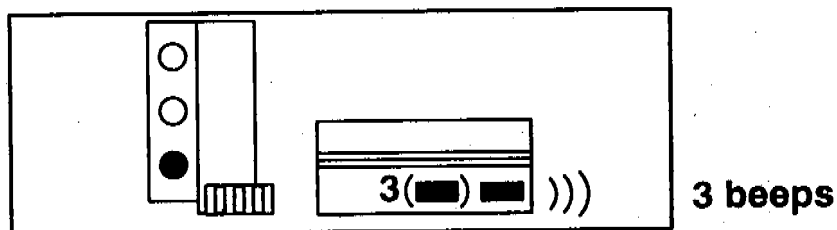
Probable causes

- There was an incomplete injection.
- The plunger or syringe carrier is operating incorrectly during injection.

Suggested actions

1. If the syringe needle is bent, check the list of actions on page 125.
2. Remove the syringe from the injector, and check the plunger for stickiness or binding. Replace the syringe if necessary.
3. Press the STOP button on the injector to clear the fault indicators. Restart the sequence.
4. If the error occurs again, obtain HP service.

Bottle in gripper



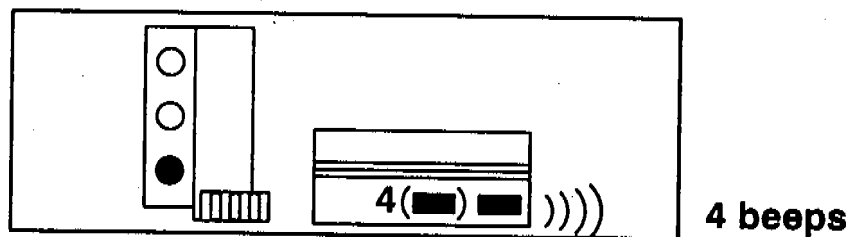
Probable cause

- The sample vial was not delivered properly and stayed in the tray gripper.

Suggested actions

1. Remove the vial, and return it to its position in the tray.
2. Ensure that the tray quadrants are snapped into place.
3. Ensure that the injector is plugged into the correct connector on the back of the controller (front or rear).
4. Check the "deliver to" location for the vial, and verify that the location is empty and free from obstructions.
5. Press the STOP button on the injector to clear the fault indicators. Restart the sequence.
6. If the error occurs again, obtain HP service.

Hard tray/injector error



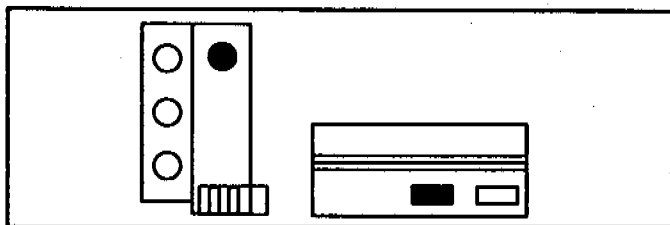
Probable causes

- The tray is not able to move.
- The injector is not responding to signals from the controller.

Suggested actions

1. Remove any obstructions from the tray that might keep the arm from moving through its full motion.
2. Press the STOP button on the injector to clear the fault indicators. Restart the sequence.
3. If the error occurs again, obtain HP service.

Align LED is On



Probable causes

- The type of turret was changed without performing the alignment procedure.
- The system was not initialized.
- There is an injector memory error.
- The injector main board was replaced.

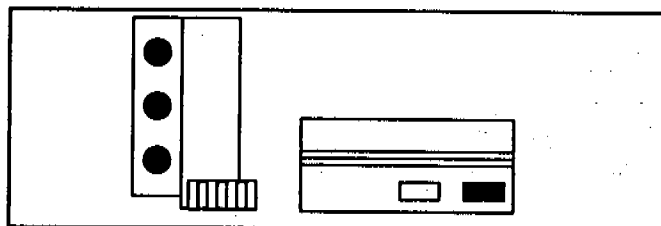
Suggested action

1. Perform the alignment procedure. (See the "Alignment procedure" in this section.)

Preventive Maintenance and Troubleshooting
Fault light symptoms

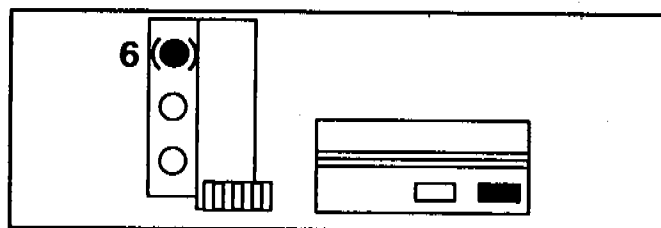
Other patterns

For any of the following patterns, obtain HP service.



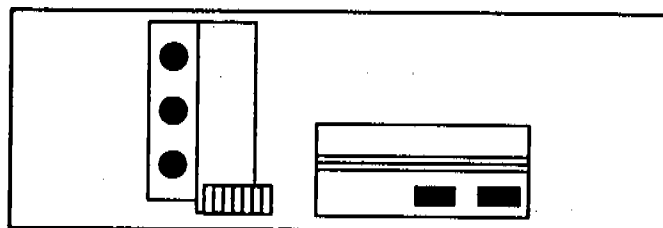
Probable cause

- There is an injector board failure.



Probable causes

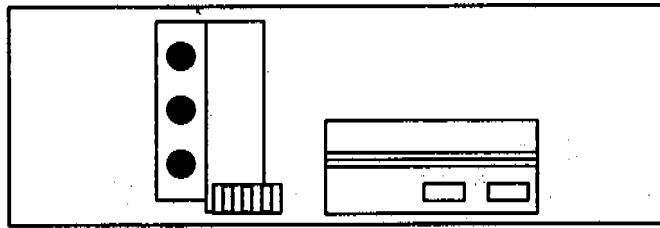
- There is an injector memory error.
- The injector main board is malfunctioning.



Probable cause

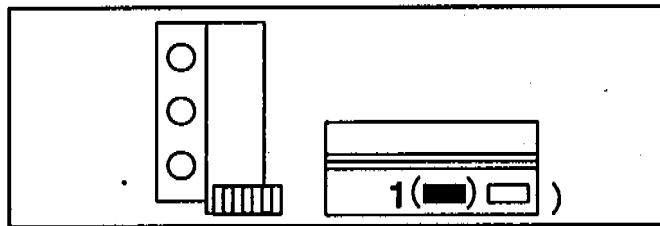
- The controller cannot be booted.

Preventive Maintenance and Troubleshooting
Fault light symptoms



Probable causes

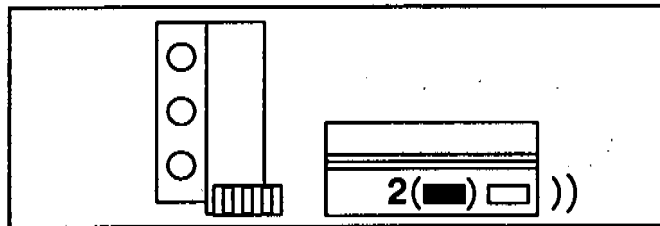
- The 18-V fuses have blown on the power supply board.
- The 18-V supply is not operable.



1 beep

Probable cause

- It failed the ± 13 V self-test.

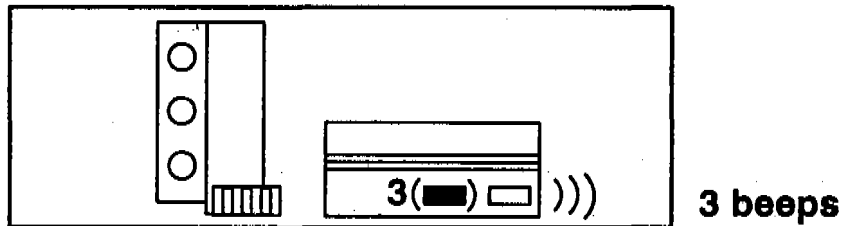


2 beeps

Probable cause

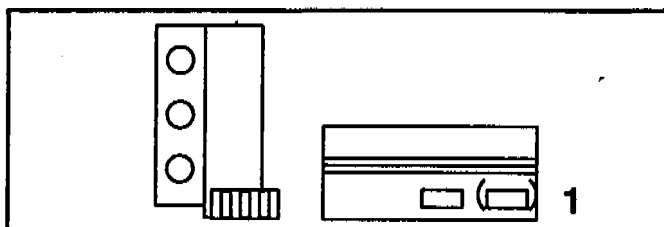
- It failed the ± 18 V self-test.

Preventive Maintenance and Troubleshooting
Fault light symptoms



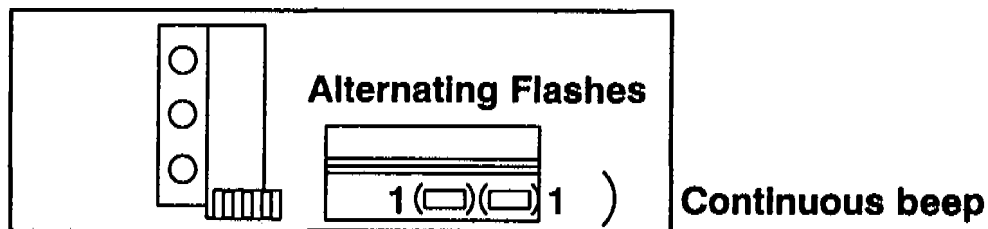
Probable cause

- It failed the fan self-test. Both fans failed.



Probable causes

- It failed the application test.
- In service mode



Probable cause

- It failed the RAM test.

Error messages

This section is an alphabetical listing of possible error messages.

BOTTLE NOT FOUND

- The gripper did not find a sample vial in the injector turret during a removal attempt.

DOWNLOAD NOT COMPLETE FILE TOO LONG

- The method file is too long.

DOWNLOAD NOT COMPLETE INVALID PARAMETER

- The value of a run parameter is not within the legal limit. Check the run parameters.

DOWNLOAD NOT COMPLETE CONFIGURATION ERROR

- The method parameters do not match the installed software or hardware.

FRONT INJECTOR COMMUNICATIONS ERROR

- There is a communications error between the front injector and the controller.

FRONT INJECTOR SYRINGE ERROR

- Go to page 141.

FRONT INJECTOR TURRET ERROR

- Go to page 142.

FRONT INJECTOR PLUNGER ERROR

- Go to page 143.

FRONT INJECTOR INCOMPLETE INJECTION

- Go to page 144.

FRONT INJECTOR MEMORY ERROR

- There is an electronics hardware problem. Obtain HP service.

FRONT INJECTOR RESET

- There is an interruption in the ± 18 V from the controller. Obtain HP service.

FRONT INJECTOR DOOR OPEN/NOT MOUNTED

- Go to page 140.

REAR INJECTOR COMMUNICATIONS ERROR

- There is a communications error between the rear injector and the controller.

REAR INJECTOR SYRINGE ERROR

- Go to page 141.

REAR INJECTOR TURRET ERROR

- Go to page 142.

REAR INJECTOR PLUNGER ERROR

- Go to page 143.

REAR INJECTOR INCOMPLETE INJECTION

- Go to page 144.

REAR INJECTOR MEMORY ERROR

- There is an electronics hardware problem. Obtain HP service.

REAR INJECTOR RESET

- There is an interruption in the ± 18 V from the controller. Obtain HP service.

Contacting Hewlett-Packard

This section contains information on how to obtain Hewlett-Packard service and how to ship or store the equipment.

Obtaining Hewlett-Packard service

To obtain service, contact your local sales or support office.

If you compile the following information before you call, Hewlett-Packard can give you better service:

- The instrument's name, model number, and serial number (e.g., HP G1513A injector module, 1111A11111). These numbers are on the unit identification label.
- A full description of the symptoms or problem.
- The maintenance agreement number(s).
- The date that Hewlett-Packard shipped the instrument to you.
- Your complete return address and telephone number.
- Your local contact: _____

| | Model No. | Serial No. | Shipped |
|------------------------------------|-----------|------------|---------|
| Controller | _____ | _____ | _____ |
| Injector (1) | _____ | _____ | _____ |
| Injector (2) | _____ | _____ | _____ |
| Tray | _____ | _____ | _____ |
| Bar Code Reader | _____ | _____ | _____ |
| Maintenance Agreement Number(s) | _____ | _____ | _____ |

Shipment or storage

Original factory or authorized Hewlett-Packard containers and packing material should be used for either shipping or storage. If other packaging materials are used, they must meet the following requirements:

1. Use a double-wall carton of at least 275 pound test.
2. The tray *must* be foam packed, and the injector's syringe carriage *must* be locked from movement. Use enough shock-absorbing material on all sides to provide a firm cushion and to prevent movement within the container.
3. Mark the outside **fragile, delicate instruments** to ensure careful handling.

If you are storing the instrument, choose a clean environment that does not exceed the following parameters:

- 95% relative humidity
- An altitude of 7,620 meters (25,000 feet)
- A temperature range of -40°C to $+75^{\circ}\text{C}$ (-104°F to 167°F)

Special Topics

Special Topics

The automatic liquid sampler allows injection onto 250- μm and 320- μm columns in addition to 530- μm columns.

This section explains how to adapt the GC and the automatic liquid sampler to use the smaller columns. Specifically, it outlines the following:

- How to install the needle into the syringe barrel
- How to check the needle-to-column size
- How to prepare the inlet
- How to use retention gaps
- How to prepare the injector
- How to replace the needle guide in the needle support foot
- How to troubleshoot 250- μm and 320- μm injections
- How to control sample vial temperatures
- How to use tray control commands

For a list of the consumables needed to perform 250 μm and 320 μm injections, see the *Analytical Columns and Supplies Catalog*.

Performing cool on-column injection onto 250- μ m and 320- μ m columns

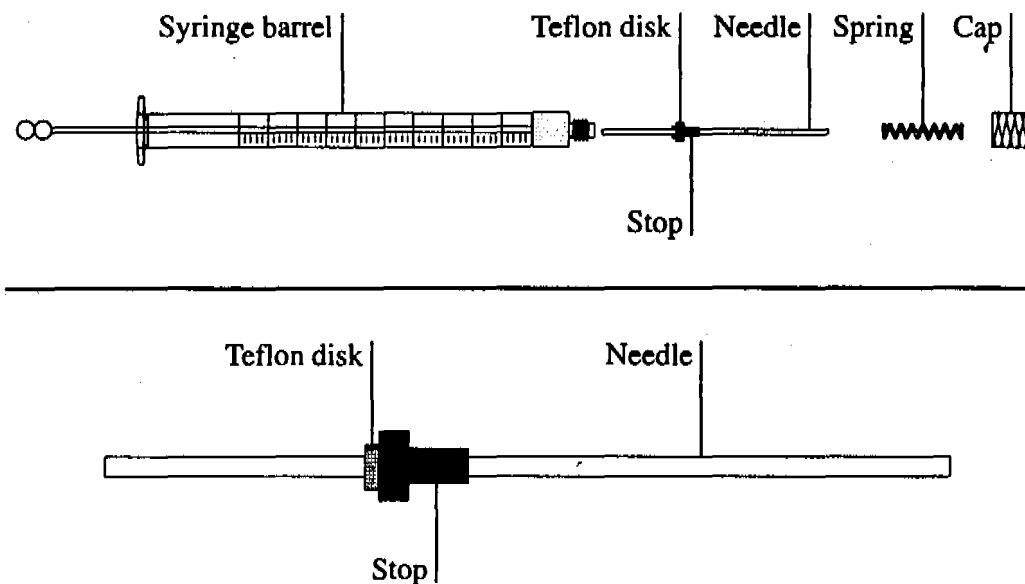
Installing the needle into the syringe barrel

The stainless steel needles used for 250- μ m and 320- μ m injections must be inserted into a glass syringe barrel (HP part no. 5182-0836 [5 μ l]). You select the correct size needle for the column you plan to use. Needles for 250- μ m injections (HP part no. 5181-7442) have silver-colored stops. Needles for 320- μ m injections (HP part no. 5181-1266) have gold-colored stops.

To insert a needle into a syringe barrel:

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

Figure 6-1

**Syringe parts and assembly.**




Checking the needle-to-column size

You need to check the needle-to-column fit because some manufacturers provide columns with internal diameters that are too small. You will bend the syringe if you try to inject into a column that is smaller than the needle.

You use the insert that is the same size as the syringe needle you will use in the analysis to verify that the column you plan to use is the correct size.

1. Identify the correct insert by the number of rings on it using the table below. The syringe needle size is listed to guide you in the choice of needles. Some of the needles listed have syringe barrels attached; some do not.

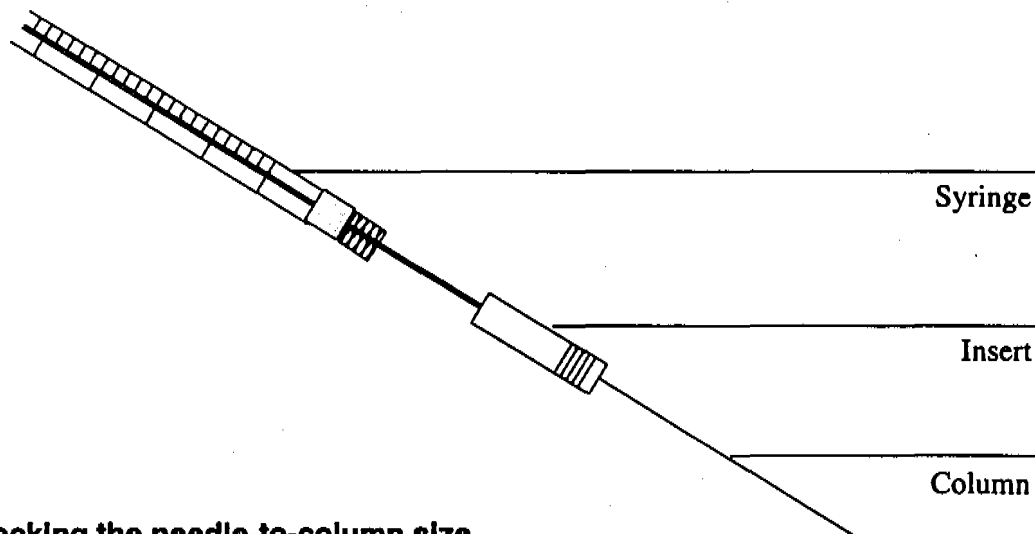
Figure 6-2

| Column Size | Insert | Syringe Needles |
|--|--|------------------------------------|
| 250 μm |  HP part no. 19245-20515 6 identification rings | 26/32 tapered |
| 320 μm |  HP part no. 19245-20525 5 identification rings | 26/32 tapered |
| 530 μm Polyimide-coated column |  HP part no. 19245-20580 0 identification rings | 23/26 tapered or 26 straight |

Consumables for automatic injection.

2. Insert the column into one end of the insert as shown in figure 6-3.

Figure 6-3



Checking the needle-to-column size.

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

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

Preparing the cool on-column inlet

To prepare the cool on-column inlet for injection into the 250- μ m and 320- μ m columns, you need to do the following:

- Change the insert and inlet septum nut.
- Align the inlet septum nut, septum, and insert.
- Install the column.
- Verify the alignment.

Changing the insert and inlet septum

The insert that you used to check the column size is installed in the inlet of the GC to guide the syringe needle into the column. The insert must correspond to the size of the column and syringe needle you will use. (See the previous section to identify the correct insert.)

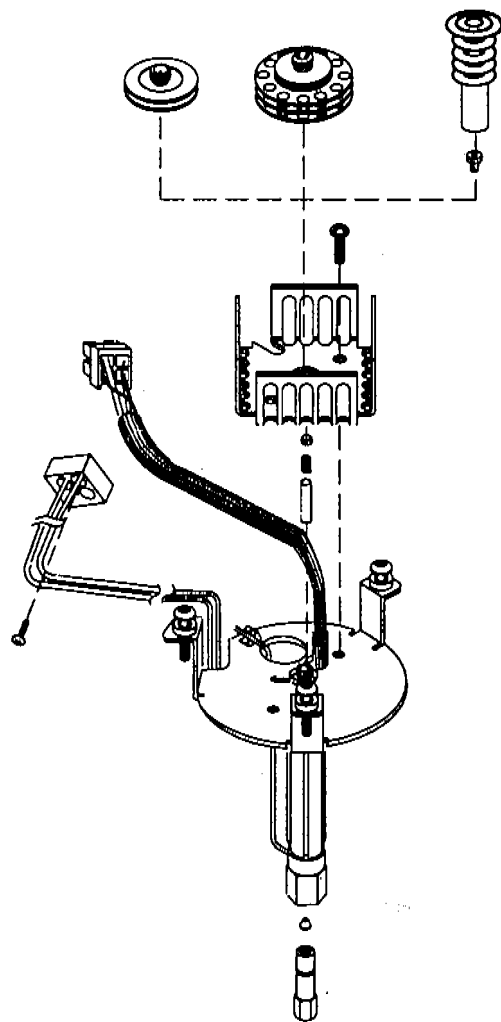
Use the following steps to remove an incorrect insert and to install a new one:

1. Lower the temperature of the GC oven. After the oven is cool, turn the oven off, and set the inlet pressure to 0.
2. Remove the column along with its column nut and ferrule.
3. On top of the oven, unscrew and remove the inlet septum nut.
4. Replace the old septum with the new through-hole septum, and set the septum nut aside.
5. Remove the spring from the inlet, and set it aside. Be careful not to lose or damage it because you will use the spring with the new insert.
6. Remove the existing insert from the inlet by pushing it out from below with a piece of column. Store the insert for possible later use.

Performing cool on-column injection onto 250- μ m and 320- μ m columns

7. Drop the new insert straight into the inlet from the top. The end of the insert in which you inserted the needle should be up; the end of the insert in which you inserted the column should be down (see the previous section on checking the needle-to-column size). Use the identification rings to help you remember which way to install the insert.
8. Replace the spring on top of the insert.

Figure 6-4



Inlet assembly.

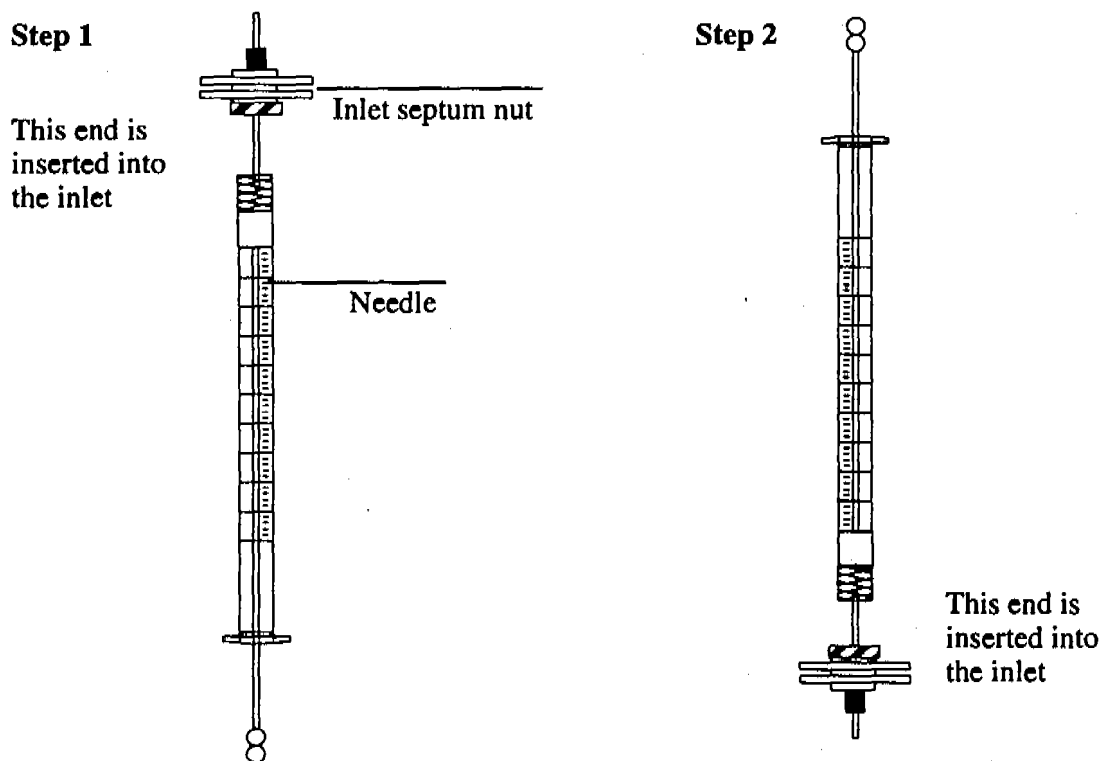
You will align and install the inlet septum nut in the next section.

Aligning the septum nut

For 250- μ m and 320- μ m on-column injections, the septum nut requires a 5-mm through-hole septum.

1. To ensure proper alignment, thread the septum and the septum nut onto a 26-gauge or larger needle or a piece of wire. The septum should rest in the cavity in the nut.
2. Turn the needle over, and insert the wire, inlet septum nut, and through-hole septum into the inlet.

Figure 6-5



Ensuring proper alignment.

3. Tighten the septum nut as tight as possible. If the septum nut is loose, the system may leak.
4. Remove the needle from the inlet.

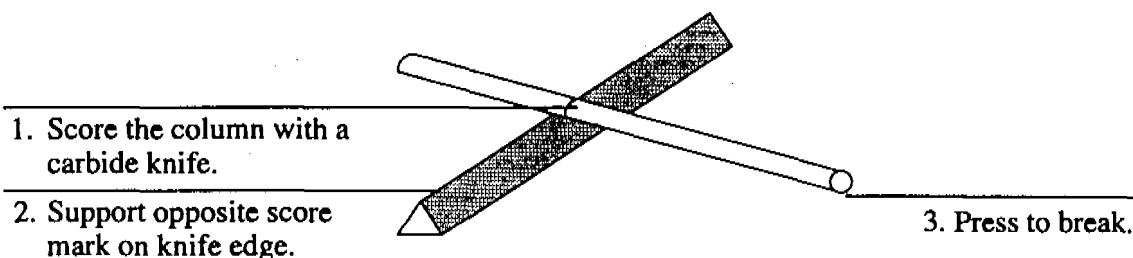
Installing a fused silica capillary column into the inlet

You must cut the column correctly to ensure effective connection. The illustrated method shown below produces the recommended square cut.

WARNING

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

Figure 6-6



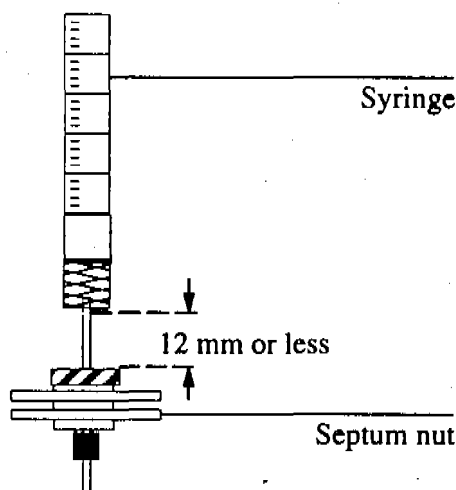
Cutting the column.

1. Slide a column nut and ferrule onto the end of the column.
2. Cut off the column end with a square cut according to the illustration above.
3. Wipe the column end with methanol to remove oil and dust.
4. Insert the column into the inlet base until it stops against the insert.
5. Finger-tighten the column nut.

Note: Make sure the column remains against the insert while you tighten the nut.

6. Use a wrench to tighten the column nut an additional quarter turn.
7. Once you have selected the syringe, verify the column installation by manually pushing it into the inlet. There should be a gap of 12 mm or less between the septum nut and the syringe barrel. If there is more than 13 mm between the syringe barrel and septum nut, your needle is not reaching the column, and you cannot perform 250- μm or 320- μm on-column injections. If this is the case, reinstall the insert and the column.

Figure 6-7



Verifying column Installation.

If you are using a retention gap, read the following section.

Using retention gaps and other precolumns

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

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

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

In general, the length of the retention gap required and type of deactivation depend on injected volume and solvent polarity. A working rule of thumb is to use 1 m of retention gap per microliter injected. For a 3- μl sample, use a 3-m retention gap. The retention gap should be wetted by

the solvent, which means it should be deactivated with a material of similar polarity. Fused silica tubing is commercially available in a range of diameters and deactivations.

Press-fit connectors

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

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

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

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

Butt connectors

Butt connectors are also popular for connecting precolumns to columns, particularly for high-temperature use. Different-size ferrules are used,

Performing cool on-column injection onto 250- μm and 320- μm columns

depending on the size of the columns. Because column ends are in contact with each other inside the ferrule during tightening, exposure to ferrule material is minimized.

Purged connectors

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

Preparing the injector

The preparation of the injector is divided into the following steps:

- How to change the needle support assembly
- How to install the syringe into the needle support assembly

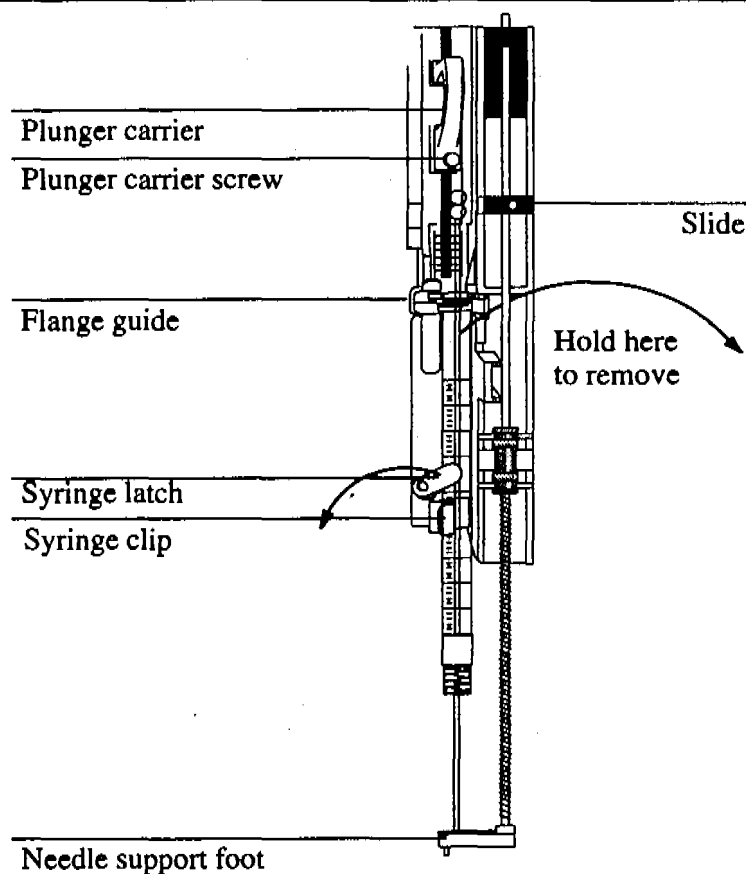
Changing the needle support assembly in the injector

When changing to 250- μm or 320- μm injection, you must change the needle support assembly. The needle support assembly is a spring-loaded rod with a needle support foot at its base. The foot supports and guides the needle during injections.

Removing the needle support assembly

1. Lay the injector module on its back on a flat surface.
2. Open the injector door.
3. Loosen the plunger screw. (The plunger screw holds the plunger in the plunger carrier.)
4. Slide the loop of the plunger carrier up as far as it will go.
5. Swing the syringe latch counterclockwise to unlock the syringe.

Figure 6-8



Removing the needle support assembly.

6. With your finger under the upper portion of the syringe barrel (just above the syringe latch), pull the syringe up, and gently remove it.

Caution

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

7. With your finger under the brass fitting of the needle support assembly, pull up gently to release the brass fitting from the syringe carriage, and remove the assembly.
8. Carefully use the brass fitting to pull the rod down until you can lift the assembly out of the syringe carriage.

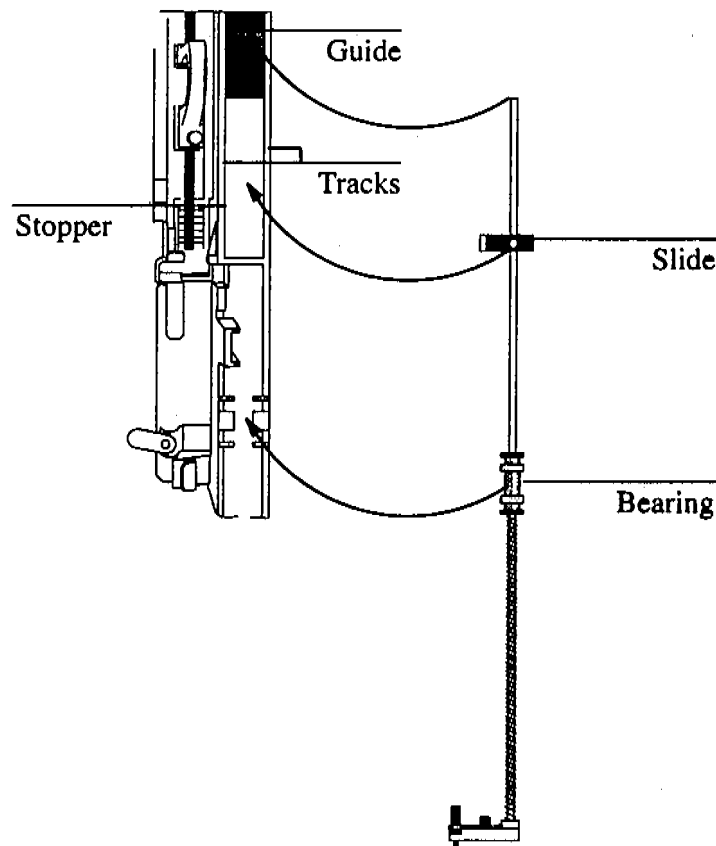
Caution

Be careful not to pull the assembly by its metal shaft. The shaft is easily bent.

Installing the 250/320- μm needle support assembly

1. Hold the new needle support assembly in your right hand, and insert the upper end of the rod into the plastic guide to the right of the plunger carrier.
2. Align the bearing on the needle support assembly with the plastic bearing clip to the right of the syringe latch.

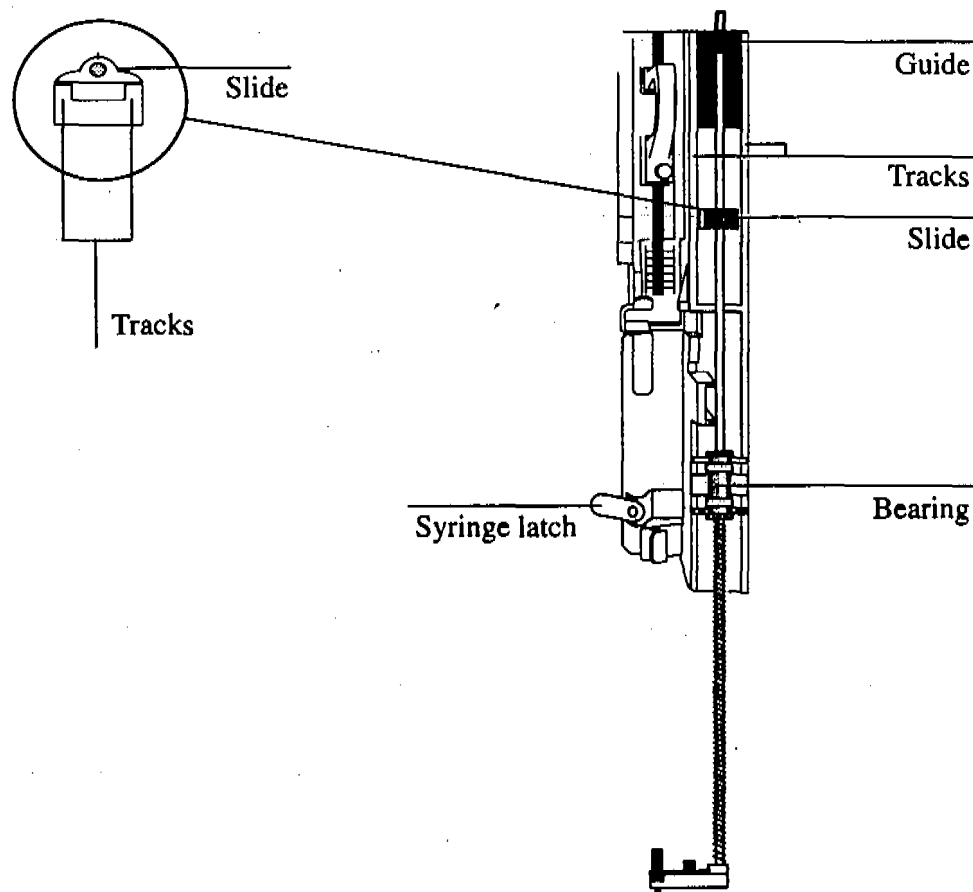
Figure 6-9



Installing the 250/320- μm needle support assembly.

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

Figure 6-10



Cross-section of slide alignment.

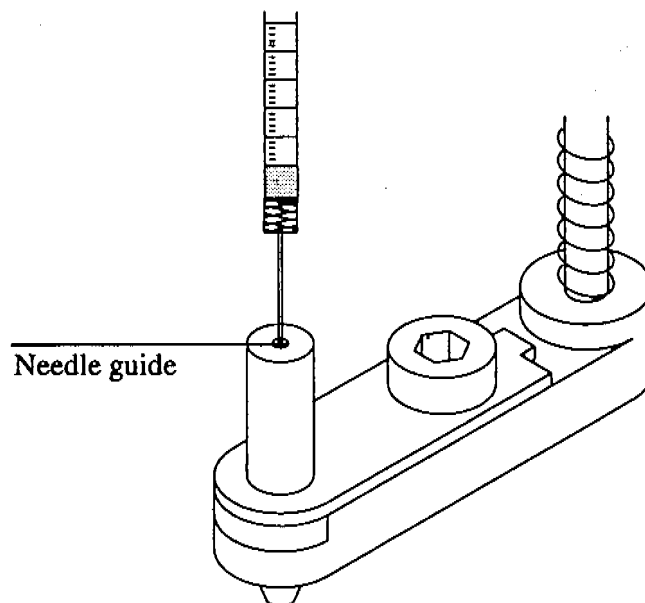
Installing the syringe into the needle support assembly

Use the following steps to install a new syringe:

1. If you have removed the injector module to install the needle support assembly, place the module back on the GC oven or on a parking post if you have one.
2. Open the injector door.

3. Pass the syringe needle through the hole of the small needle guide in the needle support foot as shown below.

Figure 6-11



Needle support foot for 250- μ m injection.

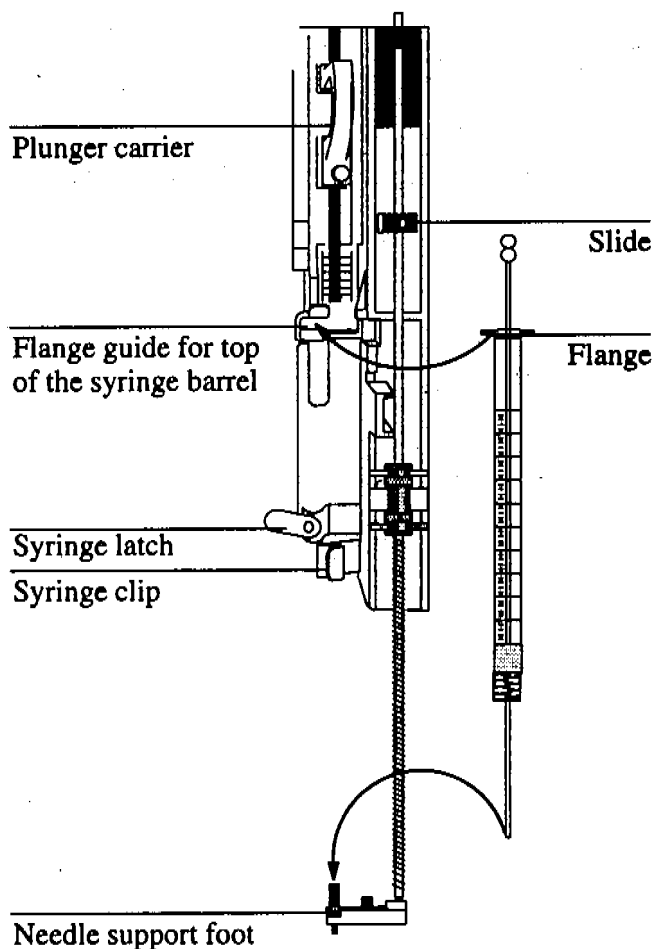
4. Align the syringe flange with the flange guide and syringe clip, and gently press the syringe into place, keeping the needle in the hole of the needle guide. Be careful not to bend the needle during this step.
5. Close the syringe latch by swinging it clockwise.

Caution

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

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

Figure 6-12



Installing the syringe for 250- μm or 320- μm on-column Injection.

Replacing the needle guide in the needle support foot

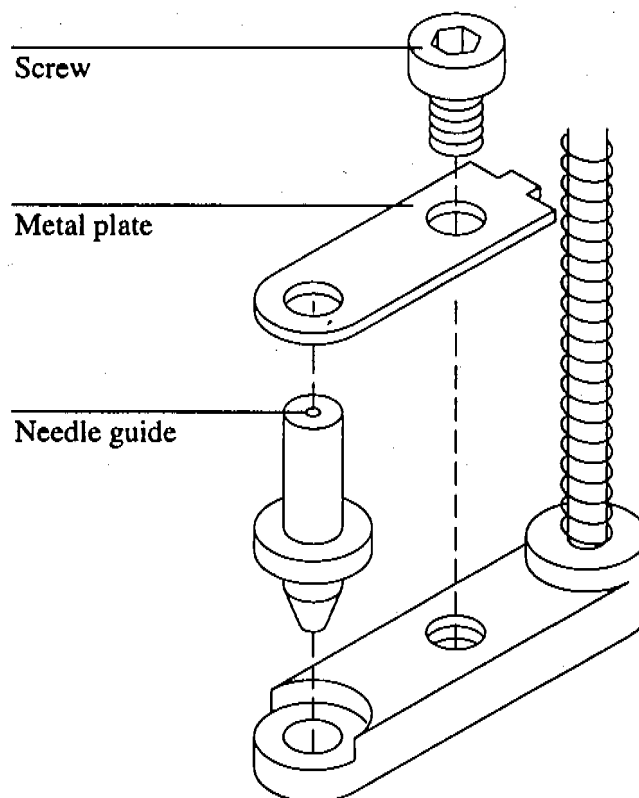
You need to check the needle guide for wear whenever you change the inlet septum. When the needle guide in the needle support foot shows signs of wear, use the following steps to replace it:

1. With an Allen wrench, remove the black screw from the needle support foot.
2. Remove the small metal plate from the foot.
3. Remove the needle guide, and replace it with a new one.
4. Replace the metal plate.

Performing cool on-column injection onto 250- μm and 320- μm columns

5. Replace and finger-tighten the screw.
6. Finish tightening the screw with a 2-mm Allen wrench.

Figure 6-13



Needle support foot for 250- μm injection.

Troubleshooting 250- μ m and 320- μ m systems

For general troubleshooting information, see chapter 5 of this manual.

| Symptom | Possible Cause | Corrective or Preventive Action |
|---|---|--|
| Bent needle | Incorrectly installed needle support assembly | Check needle support assembly installation. |
| | Defective needle | Check each syringe before installation to make sure the needle is straight. |
| | Incorrect insert | Make sure the insert is the correct size for the column and needle you use. Also check that the insert is installed correctly. |
| | Worn or damaged needle guide | Check the needle guide on the needle support foot every time you change the inlet septum, and replace if necessary. |
| | Incorrect inlet septum | Use only a 5-mm septum with a through-hole. |
| | Poor alignment of inlet septum and septum nut | Align the inlet septum and septum nut according to the instructions provided in this manual. |
| | Incorrect column internal diameter | Check the internal diameter of the column by using the appropriate insert. |
| | Closed inlet septum hole | Replace the septum. |
| No peaks or unexpectedly small peaks | Poor alignment of the inlet and the automatic injector | See the <i>HP 5890 Series II Operating Manual</i> for alignment instructions. |
| | Plugged syringe needle | Replace the needle, or clean it with wire. |
| | Worn syringe barrel | Replace the syringe often, or use a Teflon-tipped plunger. |
| | Loose removable needle | Make sure the syringe barrel caps are screwed on tightly and that the Teflon disk is in place. |
| | Incorrectly placed or missing Teflon disk on the syringe needle | Check every syringe needle to make sure the Teflon disk is present and correctly placed. |

| Symptom | Possible Cause | Corrective and Preventive Action |
|---|---|--|
| Poor precision, poor repeatability, or large standard deviation | Worn syringe barrel | Replace the syringe. |
| | Loose removable needle | Make sure the syringe barrel caps are screwed on tightly and that the Teflon disk is wrapped tightly as shown in this chapter. |
| | Widened holes in vial caps | Replace vial caps when holes widen and leaks develop. |
| | Inlet pressure set too low | Adjust the pressure. |
| | Incorrectly placed or missing Teflon disk on the syringe needle | Check every syringe needle to make sure the Teflon disk is present and correctly placed. |

Controlling sample vial temperatures

This section explains how to connect tubing to the tray quadrants, how to calculate the water bath temperature used to obtain an approximate sample vial temperature, and the pressure limitations of the quadrants.

This section does not explain how to set up a water bath and pump.

You can control the temperature of the sample vials in the tray by:

- Pumping a temperature-controlled liquid through the tray quadrants.
- Freezing an ethylene glycol and water solution inside the tray quadrants.

The quadrants are designed for low flow rates, low pressures, and long temperature equalization times. If you freeze an ethylene glycol or alcohol solution in the quadrants, fill the quadrants to less than 90% of their total capacity.

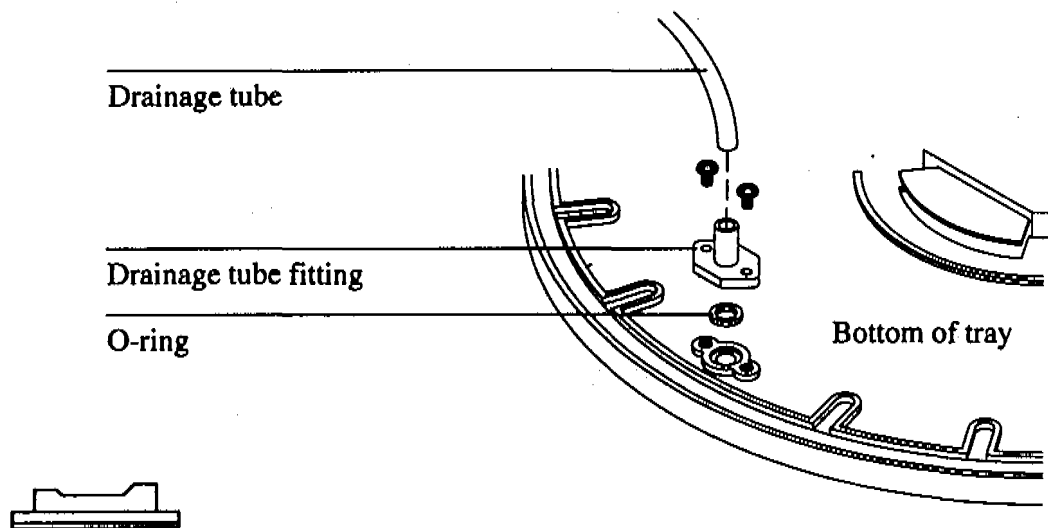
Caution

Ensure that the drainage tube is attached to the base of the tray, and remove any modules from underneath the tray. When controlling for a low sample temperature with a high ambient temperature or high humidity, water condensation from the quadrants and tray could damage equipment under the tray.

Attaching the drainage tube

Attach the drainage tube fitting and drainage tube to the bottom of the tray. The hardware is packaged with the tray.

Figure 6-14

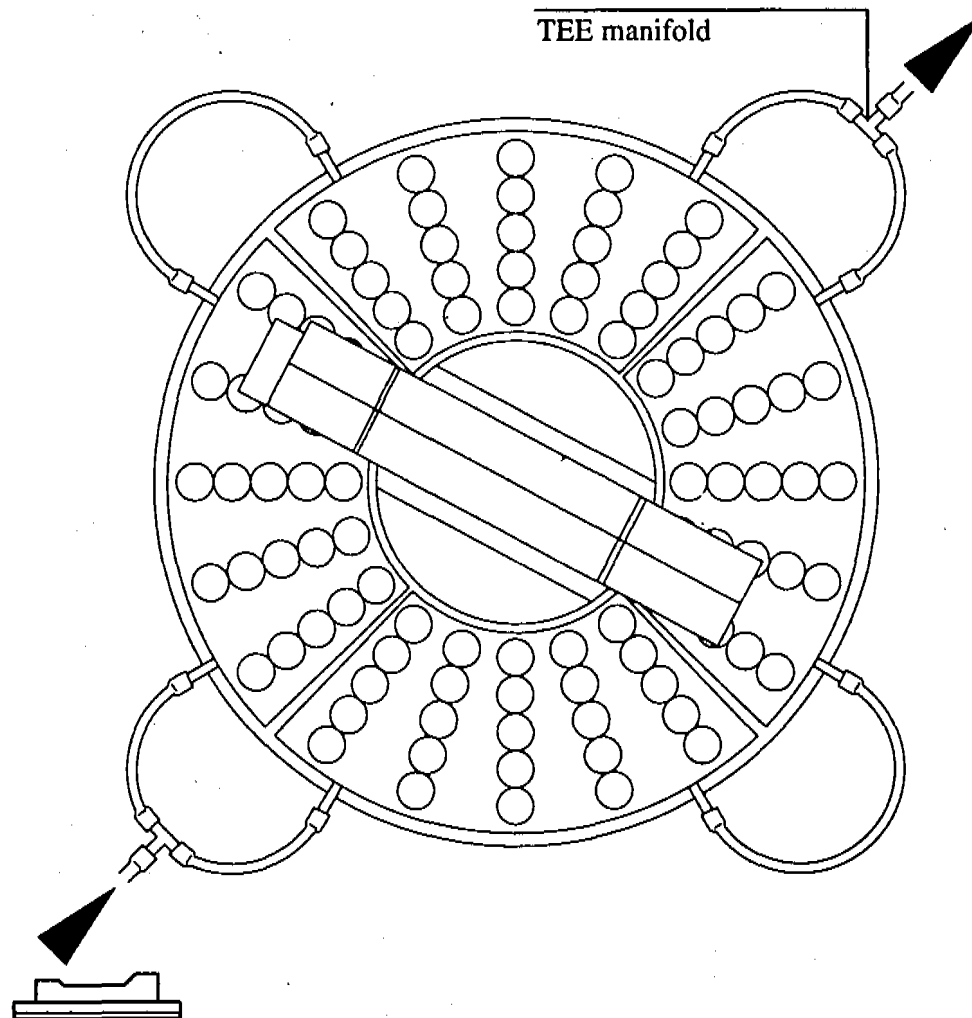


Attaching the tray's drainage tube fitting.

Connecting the tray quadrants

The recommended plumbing used to connect the tray quadrants is shown in figure 6-15. Use 6.35-mm id (1/4-in.) tubing.

Figure 6-15



Tray quadrant plumbing.

Calculating the water bath temperature

The temperature of the sample vials depends on several factors, including the ambient temperature of the air around the tray and the temperature of the water bath.

Caution

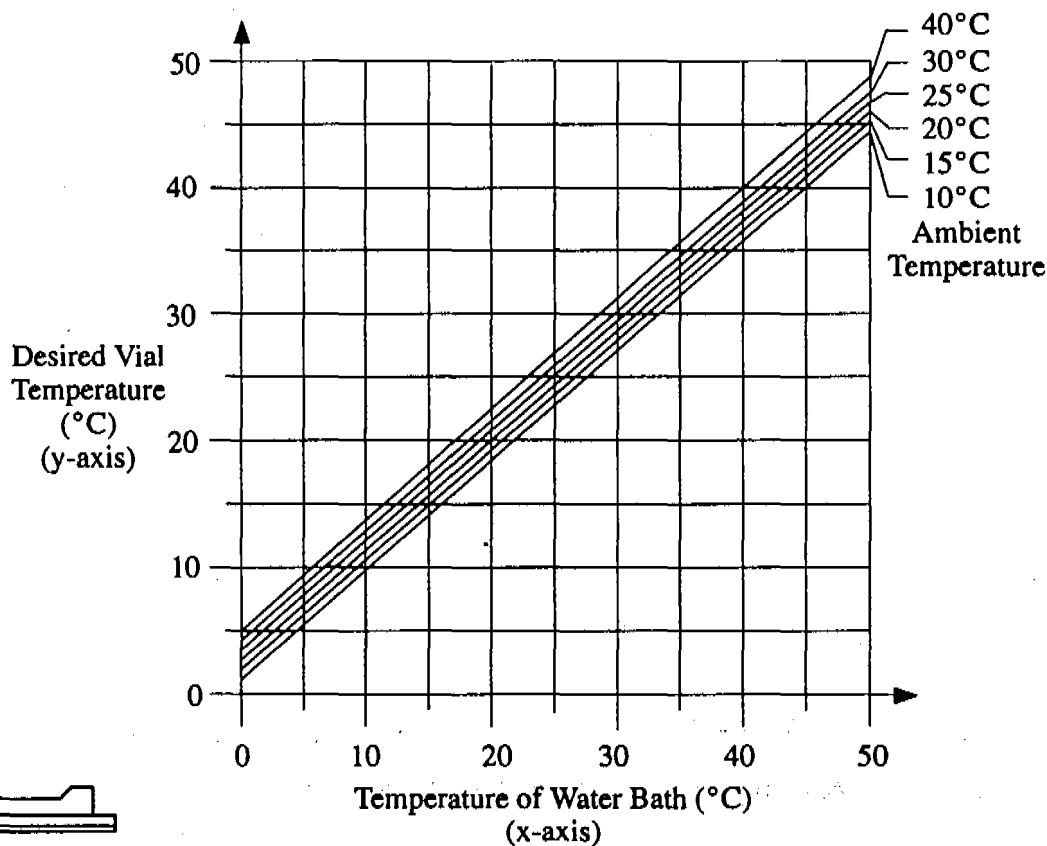
If the sample temperature is critical for accuracy, experiments should be performed to validate water bath settings. Actual heat transfer varies with vial material, humidity, condensation on the vial, and flow rates.

Performing cool on-column injection onto 250- μm and 320- μm columns

To calculate the temperature needed for the water bath, complete the next four tasks. To convert temperature units, use the formula $^{\circ}\text{C} = 5/9 (^{\circ}\text{F} - 32)$ or $^{\circ}\text{F} = 9/5(^{\circ}\text{C}) + 32$.

1. Measure the average ambient air temperature near the tray.
2. Find the desired sample vial temperature on the y-axis of the graph below.
3. From the point you selected in step 2, draw a horizontal line over to the diagonal line that corresponds with the ambient air temperature you measured in step 1.
4. From the intersection of desired vial temperature and ambient temperature, draw a vertical line down to the x-axis of the graph, and read the temperature needed for the water bath.

Figure 6-16



Sample vial temperature versus water bath temperature for values of ambient air temperature.

Tray quadrant temperatures and pressures

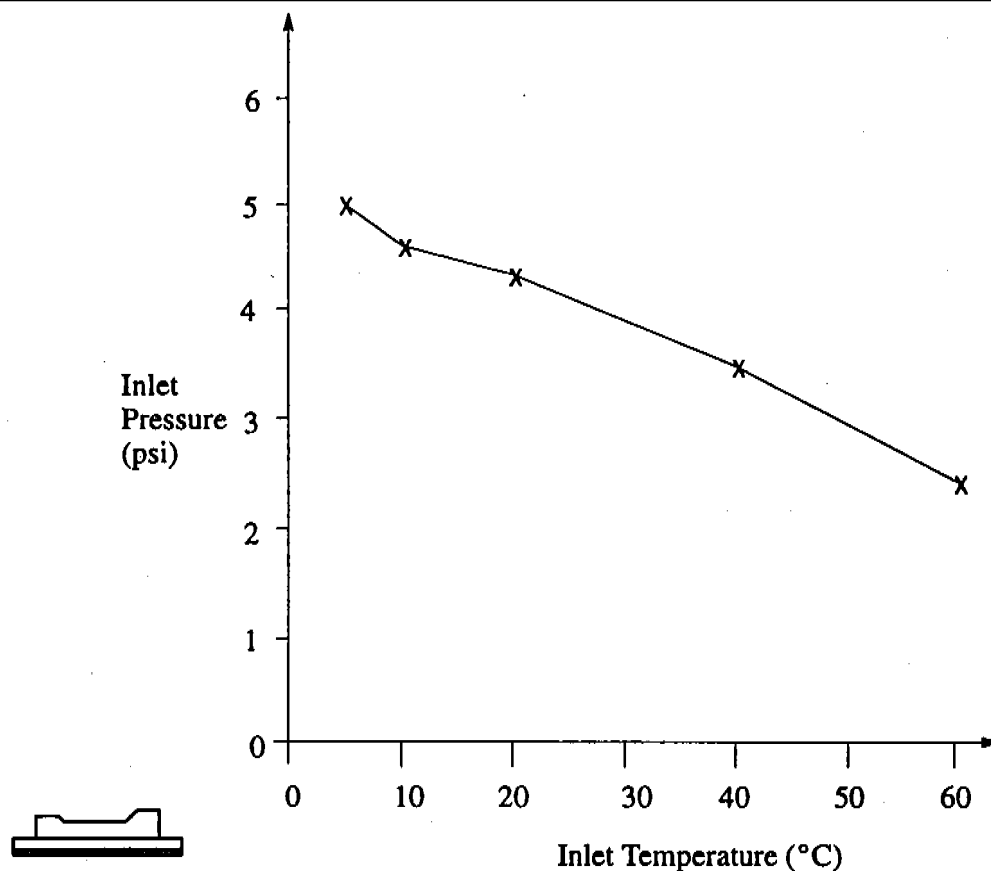
Figure 6-17 shows the maximum pressures and temperatures measured at the output to the quadrant plumbing.

For example, with a water bath temperature of 40°C, the output pressure to the tray quadrants should not exceed 3.5 psi.

Caution

Do not allow the inlet pressure and inlet temperature to exceed these limits. If you operate the bath outside this range, the quadrants could be damaged and cause sample vial delivery problems.

Figure 6-17



Maximum Inlet pressures and temperatures.

Water bath and pump specifications

The water bath and pump system used to control the sample vial temperatures should meet the following specifications.

- Safety requirements for the components must meet national standards, be suitable for unattended operation, be suitable for continuous operation, and be controllable for high-temperature protection.
- The minimum cooling power is 100 W at bath temperatures of 5°C.
- If you use a built-in pump, it must be suitable for external circulation of liquid and for connection of 6.35-mm id (1/4-in.) tubing.
- If you use a pressure pump, it must maintain a pressure between 1.5 and 2.5 psi.
- If you use a suction pump, the pump vacuum cannot exceed -4 psi.
- Use distilled water plus a maximum of 3% antifreeze.

Tray control commands

Programming devices can be used to tell the tray where to deliver and retrieve sample vials. The most common configuration is a device that is part of the Instrument Network (INET) and uses BASIC as a programming language. For example, you can use an HP 3396 integrator with BASIC to send special commands to the tray.

This section contains some general rules about using tray control commands, a description of their syntax, and an example of how to use them with a bar code reader.

General rules

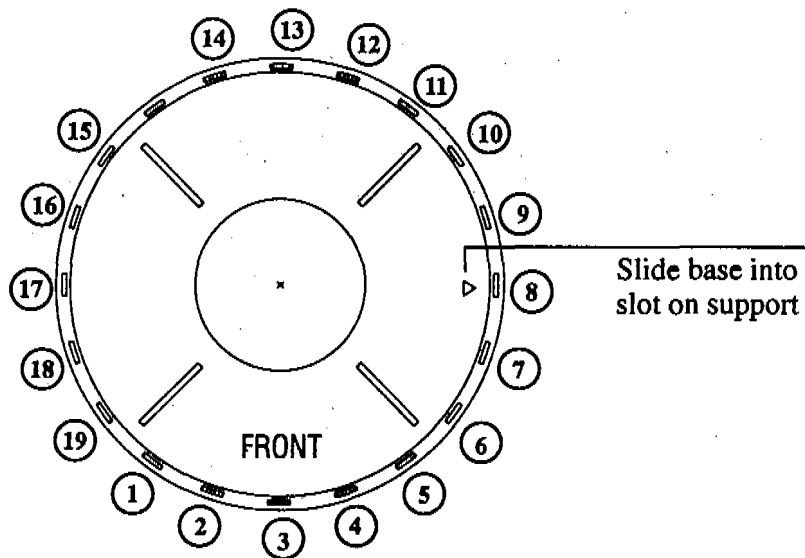
- At least three letters of the tray commands must be used. Additional letters, up to the full length of the word, may be used for improved readability. For example, the MANUAL command may be entered as MAN, MANU, MANUA, or MANUAL.

- Unlike other setpoints in the automatic liquid sampler, manual sequencing mode is replaced with the normal sequencing mode after the power is turned off.
- It is recommended that the turrets not be left in an energized state longer than necessary to minimize power consumption and temperature changes. Also, if the turret is not returned to position 0 before the next injector operation (either an injection sequence or a MOVE, RETURN BOTTLES, or TURRET command), then there will be an additional delay while the turret goes through a homing operation.
- The firmware in the controller evaluates your commands. If there are no syntax errors, the tray responds with A NO ERRORS. If there are syntax errors, the tray responds with A and a description of the error.

Syntax and descriptions

- MOVE x,y Picks up the bottle in position x and places it in position y. Positions x and y may be:
- Numbers in the range 1 to 100, referring to bottle positions in the sample tray.
 - FRONT or REAR, referring to position 1 in the front or rear injector turret.
 - EXT1 through EXT19, referring to positions in a circle beyond the outer row of bottles in the tray. Figure 6-18 maps these locations.

Figure 6-18



Map of locations on outer row of the tray.

MANUAL SEQUENCE

The MANUAL SEQUENCE command bypasses the normal sample sequencing performed by the automatic liquid sampler. Once manual sequencing mode has been activated, it is possible to manipulate sample bottles, moving them to and from the injectors with the MOVE command, and then inject from the bottles currently in the injectors simply by starting a sequence. In manual sequencing mode, the injections/bottle first bottle and last bottle setpoints are all ignored, and each injector will make just one injection but only if a bottle has been moved to its turret.

AUTO SEQUENCE

The AUTO SEQUENCE command returns the automatic liquid sampler to normal sequencing mode after being placed in manual mode.

RETURN
BOTTLES

The RETURN BOTTLES command is a quick way to return bottles that have been moved out of the tray back to the positions where they originated. Bottles that are already in the tray are not affected even if they have been moved there from somewhere else. If a bottle has been in two or more positions within the tray, then only the most recent one will be remembered, and that is the one to which it will be returned. This command is also very useful if an error has occurred or a sequence has been aborted, and it is not known exactly where all the bottles are presently located.

TURRET x,y

This command rotates the front injector turret to position x and the rear injector turret to position y.

The TURRET command controls the positioning of the injector turrets. Normally at the end of any injector operation, the sample turret is returned to its rest position with its motor deenergized so there is only a small residual holding torque to keep it in that position. By using the TURRET command, it is possible to move the turret to any one of ten other positions and leave the motor energized to provide a high-holding torque.

The turret position values may be any integer from 0 to 10. When either value is omitted, that turret is not affected by the command. If both are omitted, the command performs no function. Position values 1 to 10 correspond to ten equally spaced turret positions at 36-degree intervals. The motor is left energized after the turret is moved to any of these positions. The position values 1 to 5 position the turret with sample positions 1, 2, and 3, A WASH, and A WASTE, respectively, facing directly toward the front of the injector.

Performing cool on-column Injection onto 250- μ m and 320- μ m columns

Position 0 is the rest position of the turret. When an injector is commanded to go to this position from one of the other ten positions, it takes the number of motor steps that should be required to get there and makes sure that it is at the right position. Then it deenergizes the motor. In this way, it is possible to check that no motor steps were lost in going to a turret position and back.

RESET The RESET command clears error conditions in the injectors and tray. Often after an error has occurred, an error light will flash an error code and the automatic liquid sampler will not become ready for the next injection. Sending the RESET command will correct this condition.

Example program with a bar code reader and tray

This program illustrates the following:

- Using bar code reader commands to read a bottle label
- Examining the bar code reader message for a specific character and agitating the sample if it is found
- Making the sample injection

The program simulates a sequence by repeating these actions for a range of tray positions. It uses the INET_IO command from HP 3393A or HP 3396 BASIC to transmit commands and receive responses. The same thing could be done with a short subroutine that directly controls a port.

Note the use of a dummy sequence (line 330). This sequence is set up for one injection of one sample; the program uses it repeatedly as it steps through the range of bottle numbers. The sequence is stored on a disk and loaded when required. See the integrator and BASIC manuals for more detail.

Special Topics

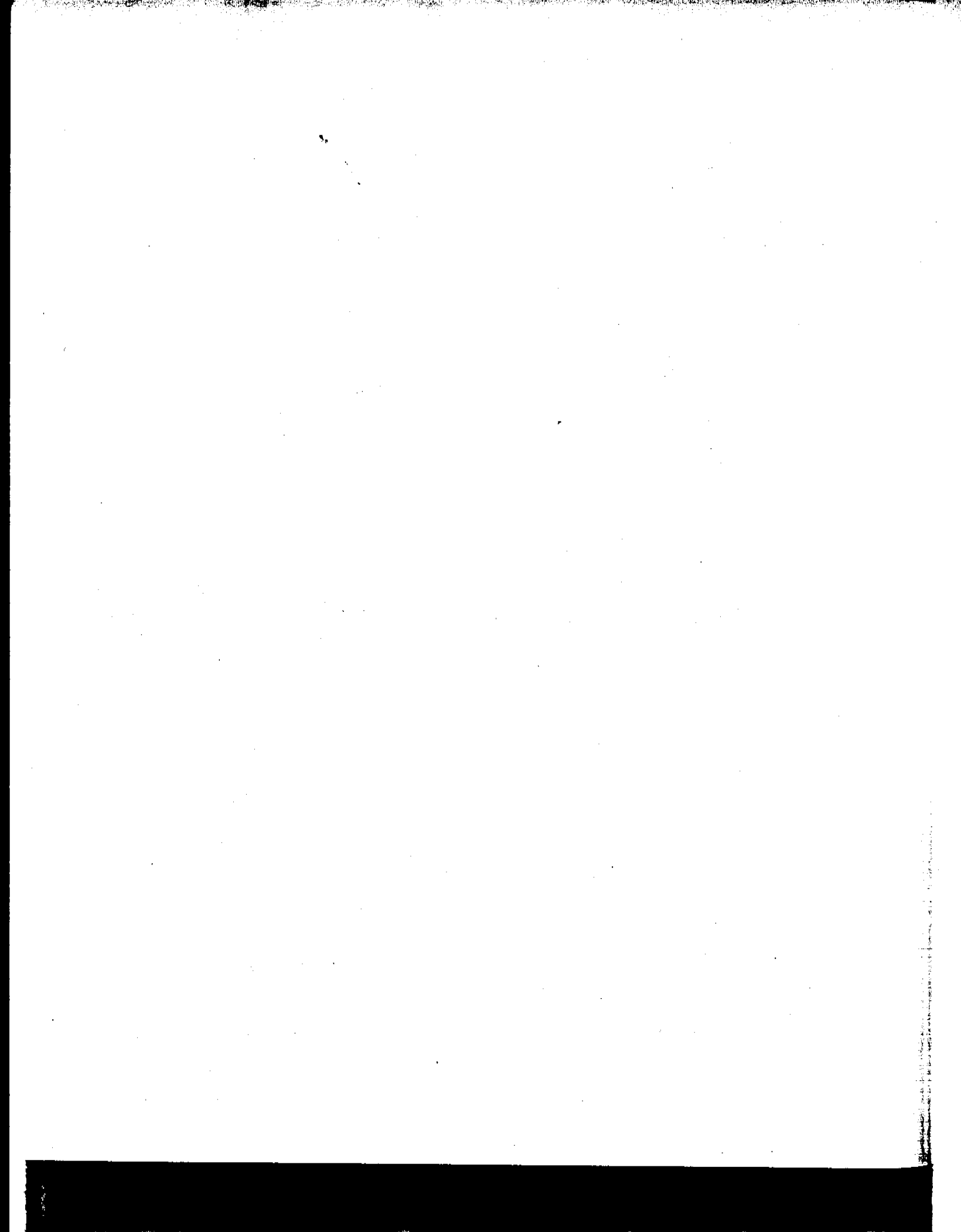
Performing cool on-column injection onto 250- μ m and 320- μ m columns

```
10 PRINT "USING THE BAR CODE READER IN AN ANALYTICAL SYSTEM"
20 PRINT
30 PRINT "THE LABEL FORMAT IS 14 CHARACTERS MAXIMUM."
40 PRINT
50 PRINT "IF A + SYMBOL APPEARS ANYWHERE ON THE LABEL, THE"
60 PRINT "SAMPLE WILL BE AGITATED BEFORE INJECTION."
70 PRINT
80 PRINT "IF THE SYSTEM CANNOT READ THE LABEL OR IF THE VIAL IS NOT"
90 PRINT "LABELED, IT WILL BE RETURNED TO ITS ORIGINAL POSITION."
100 PRINT
110 PRINT "THE USER MUST SUPPLY THE SAMPLER ADDRESS AND THE"
120 PRINT "NUMBERS OF THE FIRST AND LAST BOTTLES TO BE INJECTED."
130 PRINT
140 REM ***** DIMENSION STRING VARIABLES
150 DIM RESP$(40)  ! RESPONSE STRINGS THAT MATTER
160 DIM JUNK$(40)  ! RESPONSE STRINGS THAT DON'T MATTER
170 DIM BARCODE$(20) ! THE BAR CODE DATA, ENCLOSED IN QUOTES
180 REM
190 REM ***** ENTER THE SAMPLER INET ADDRESS
200 PRINT
210 PRINT
220 PRINT "ENTER THE INET ADDRESS OF THE SAMPLER."
230 INPUT AD
240 REM
250 REM ***** SET THE AUTOMATIC LIQUID SAMPLER TO MANUAL OPERATION
260 INET_IO AD,"MAN",JUNK$
270 REM
280 REM ***** SET UP THE VIAL SEQUENCE
290 PRINT "ENTER THE FIRST VIAL NUMBER:"
300 INPUT FIRST
310 PRINT "ENTER THE LAST VIAL NUMBER:"
320 INPUT LAST
330 IF LAST >= FIRST THEN GETSEQ "A:DUMMY" ELSE STOP
340 REM
350 REM ***** BEGIN SEQUENCE LOOP
360 FOR I=FIRST TO LAST
370 INET_IO AD,"RESET",JUNK$
380 INET_IO AD,"MOVE"&STR$(I)&" ,EXT3",JUNK$
390 INET_IO AD,"READ",RESP$
```

Special Topics

Performing cool on-column injection onto 250- μ m and 320- μ m columns.

```
400 REM
410 REM ***** FIND THE BEGINNING OF THE BAR CODE DATA
420 CODE=POS(RESP$,"=")+1
430 BARCODE$=RESP$(CODE:)
440 REM
450 REM ***** CHECK FOR A VALID BAR CODE
460 IF BARCODE$="" THEN GOSUB INVAL_BC
470 IF BARCODE$ <> "" THEN GOSUB VALID_BC
480 NEXT I
485 INET_IO AD,"AUTO",JUNK$
490 PRINT
500 PRINT
510 PRINT "END OF DEMO -- THANKS"
520 END
530 REM
540 REM *****SUBROUTINES FOLLOW
550 VALID_BC:
560 PRINT
570 PRINT "===== "
580 PRINT "  BEGIN ANALYSIS"
590 PRINT "    "&RESP$
600 PRINT "===== "
610 PRINT
620 IF POS(RESP$,"+") <> 0 THEN INET_IO AD,"AGIT",JUNK$
630 INET_IO AD,"MOVE EXT3,FRONT",JUNK$
640 START SEQ_NOW
650 INET_IO AD,"MOVE FRONT",&STR$(I),JUNK$
660 PRINT "===== "
670 PRINT "  END ANALYSIS"
680 PRINT "    "&RESP$
690 PRINT "===== "
700 RETURN
710 REM
720 INVAL_BC:
730 PRINT
740 PRINT "*****"
750 PRINT "INVALID OR MISSING LABEL ON "&RESP$
760 PRINT "*****"
770 PRINT
780 PRINT
790 INET_IO AD,"MOVE EXT3",&STR$(I),JUNK$
800 RETURN
```



Glossary

Glossary

angular axis: the direction describing the rotation (clockwise and counterclockwise) of the tray's arm. The tray uses this motion to place the vial gripper above a group of five vials or in the proper orientation to the bar code reader or injector turret. It also uses this motion to engage or disengage the vial gripper's claw around the neck of the vial. See also **home positions**.

bleed: see **septum bleed**.

bottle: the 4 ml glass bottles placed in the injector turret to hold solvent or waste. See **vial**.

capillary column: modern capillary columns are divided into two classes: high resolution and low resolution. The low resolution columns have internal diameters of 0.4 mm to 0.75 mm. The high resolution columns have internal diameters of 0.1 mm to 0.4 mm.

configuration: the specific arrangement of automatic liquid sampler modules, gas chromatograph, and data handling devices to operate as a system.

cool on-column: an injection technique which places all of the sample directly onto the column without vaporization.

fast injection: a patented method of introducing a sample to a heated inlet without the negative affects of needle fractionation.

If you are using the automatic liquid sampler for the first time, you may not be aware of the changes you might see in the resulting chromatograms. With automated fast injection, you obtain chemical performance that is not available with manual injection. Most of the changes are due to:

- Eliminating the variation inherent in manual injection including the injection volume, the start time, and the amount of time the syringe needle is in the heated inlet.

- Reducing the amount of sample that boils out of the needle when injecting into heated inlets.

Reducing the amount of vaporization from the needle may result in unexpected changes in your chromatogram.

- The peak areas of your chromatograms may be smaller. Automatic fast injection delivers the desired setpoint volume of sample.

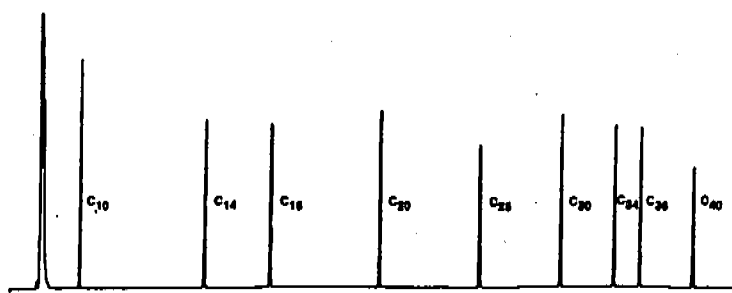
Without fast injection, residual amounts of sample boil out of the needle and enter the inlet. This extra amount could measure up to 1 μ l.

- The peak areas of your chromatograms may show less differentiation between the low boiling and high boiling components.

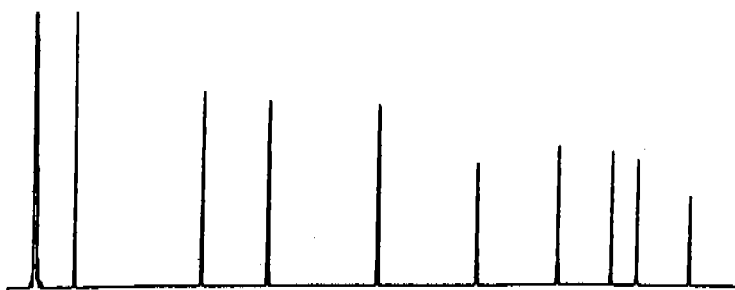
Without fast injection, the sample you introduce is richer in low boiling components than high boiling ones because of fractional distillation in the needle. Not only does residual sample in the needle enter the inlet, but the low boiling components boil off first. This is called needle fractionation or discrimination.

The following chromatograms compare manual injection with automatic fast injection from the automatic liquid sampler for a 1 μ l sample of C10 to C40 in hexane.

Automated Injection



Manual Injection



For more information on the performance of the automatic liquid sampler, order the following technical papers from your HP representative:

Publication No. 43-5953-1843: Snyder, W. Dale. Fast Injection with the HP 7673A Automatic Injector: Chemical Performance, Technical Paper 108, June 1985.

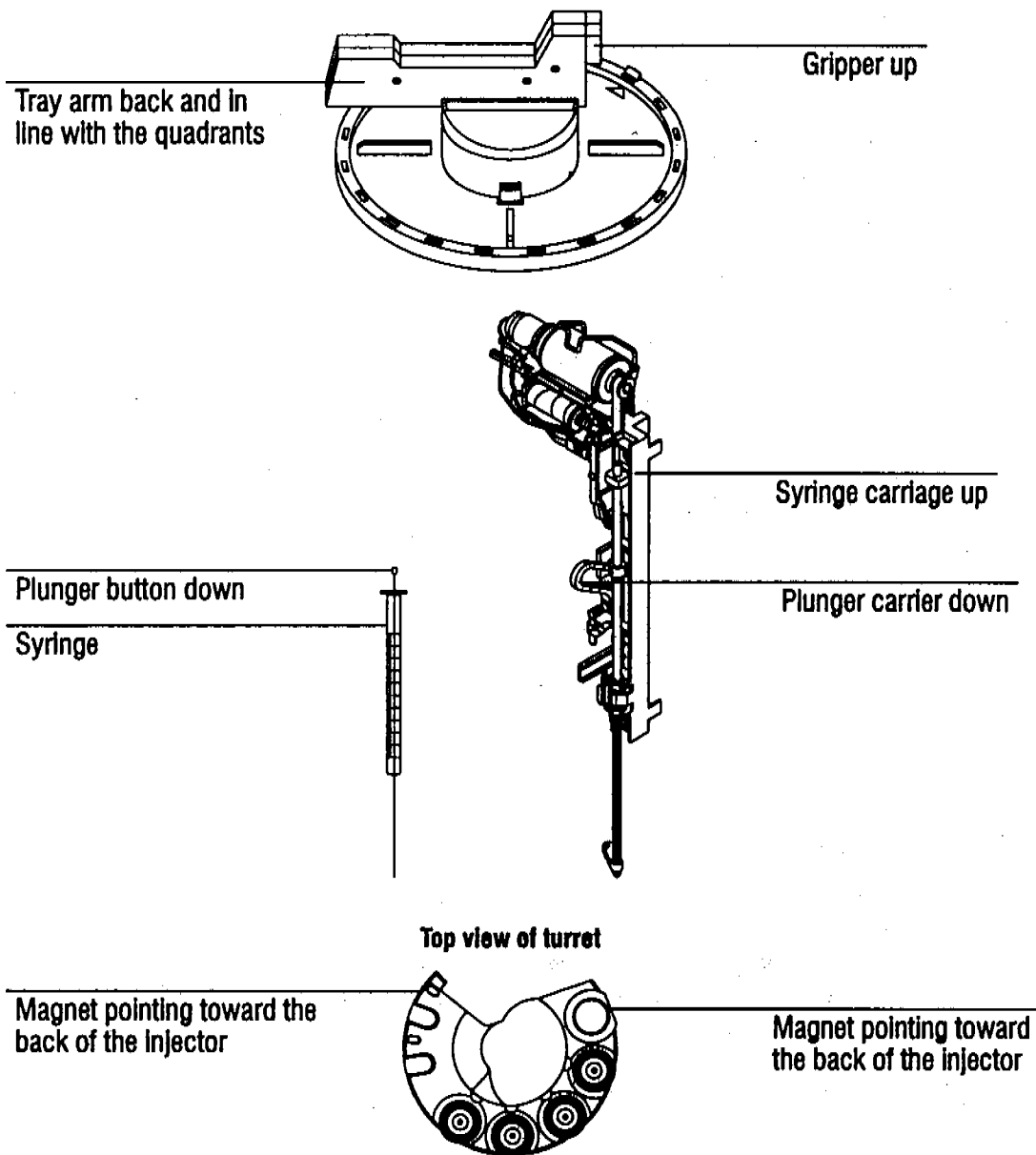
Publication No. 43-5953-1878: Snyder, W. Dale. Performance Advantages of the HP 7673A Automatic Injector Over Manual Injection, Technical Paper 109, August 1985.

Publication No. 43-5953-1879: Kolloff, R. H. C. Toney, and J. Butler. Automated On-Column Injection with Hewlett-Packard 7673A Automatic Injector and HP 19245A On-Column Capillary Inlet - Accuracy and Precision, Technical Paper 110, August 1985.

gripper axis: the direction describing the vertical (up and down) movement of the tray's vial gripper, sometimes called the z-axis. The tray combines movement along the zeta axis and the theta axis to pick up and drop off vials to specific locations on the tray or injector turret. See also **home positions**.

ghost peaks: small peaks that usually indicate that contamination is entering the system.

home positions: each of the moving parts has a reference point from which it moves to pick up, deliver, or inject samples. The parts move to their home positions between each run, when power is turned on, and when a run is started. The following illustration shows the home positions.



homing: the process of the turret, syringe carriage, syringe plunger, vial gripper, and tray arm moving to their home positions.

INET: an acronym which stands for *instrument network*.

injection: the injector motion that delivers the sample to the inlet or column of the gas chromatograph.

injection port: see **inlet**.

inlet: the terms **inlet** and **injection port** are used interchangeably in this manual.

interaction: the attraction or repulsion between two chemical species in a specific chemical environment. For example, some sample components interact with a glass inlet liner unless the liner is deactivated.

LED: an acronym which stands for *light-emitting diode*.

list: to present the contents of a program or file in readable form, often a printed copy or displayed on an integrator chart or computer screen.

nanoliter injection: an adapter to the injector that enables injection volumes from 0.1 μl to 4.0 μl .

needle fractionation: the boiling off of sample in the syringe needle during injection. Not only does residual sample in the needle enter the inlet, but the low boiling components boil off first. See **fast injection**.

parameter: an element, sometimes optional, that provides additional information to a command.

peak area discrimination: used to describe a chromatogram with peak areas that are not reproducible.

prompt: a message or symbol requesting you to enter information. For example, the run parameter prompts of the HP 3396A Integrator when you are editing a sequence.

radial axis: a direction describing the horizontal (forward and backward) movement of the tray's arm. The tray uses this motion to position the vial gripper at the correct distance from the center of the tray. See also **home positions**.

retention time: the time it takes from the moment of injection until a compound elutes and is detected at the peak maximum.

run: a discrete analysis performed under a given set of conditions on one instrument.

run time: the time, during the run, that has elapsed from the initiation of data acquisition (or time of injection). For completed runs, this term refers to the total time from injection (or initiation of data acquisition) to the end of the run (or termination of data acquisition).

sample: a liquid, gas, solid, or heterogeneous portion of material that is representative of the whole.

sample carryover: any memory of the previous sample shown in the chromatogram.

After an injection, the syringe retains a volume of sample in the needle and between the plunger and barrel that ranges from 0.6 to 1 μl . Normal laboratory practice requires washing the syringe with a solvent and/or the next sample to dilute and wash away the remaining sample.

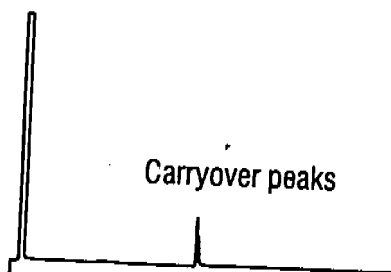
The first two chromatograms show the effect of carryover when 1 μl from a vial of methanol is injected after 1 μl from a vial of a solute dissolved in methanol. The peaks in the second chromatogram are from the solute left in the syringe from the first injection.

The third chromatogram shows the result of washing the syringe with four solvent washes. The carryover peaks disappear.

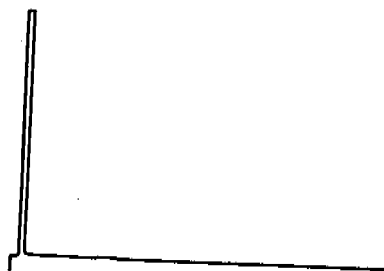
Sample #1: 20 mg/ml of solute in methanol



Sample #2a: Methanol blank without washes



Sample #2b: Methanol blank after 4 washes



septum bleed: the inherent baseline noise level emanating from a column due to decomposition or volatilization of low molecular weight products mainly from the stationary phase. The septum is another source of volatiles that cause noise.

septum life: usually refers to the number of injections a septum survives before it begins to leak.

sequence: a set of instructions that defines how a piece of equipment, for example an integrator, performs more than one automated run. These instructions usually include the automatic liquid sampler parameters, instrument equilibration time, method name, and a sample information table. Note that sequences can be recursive, that is, one sequence can contain another sequence.

solute: the components present within a given sample.

solvent: usually, the main component of a sample mix.

split injection: an injection technique which allows only a portion of the sample to be routed into the column. The rest of the sample is vented. This technique compensates for the low tolerance which high resolution capillary columns have for concentrated samples.

splitless injection: an injection technique which directs all of the sample onto the column after it has vaporized in the injection port.

standalone: the controlling method associated with using the injector's own electronics to set the run parameters and operate the automatic liquid sampler.

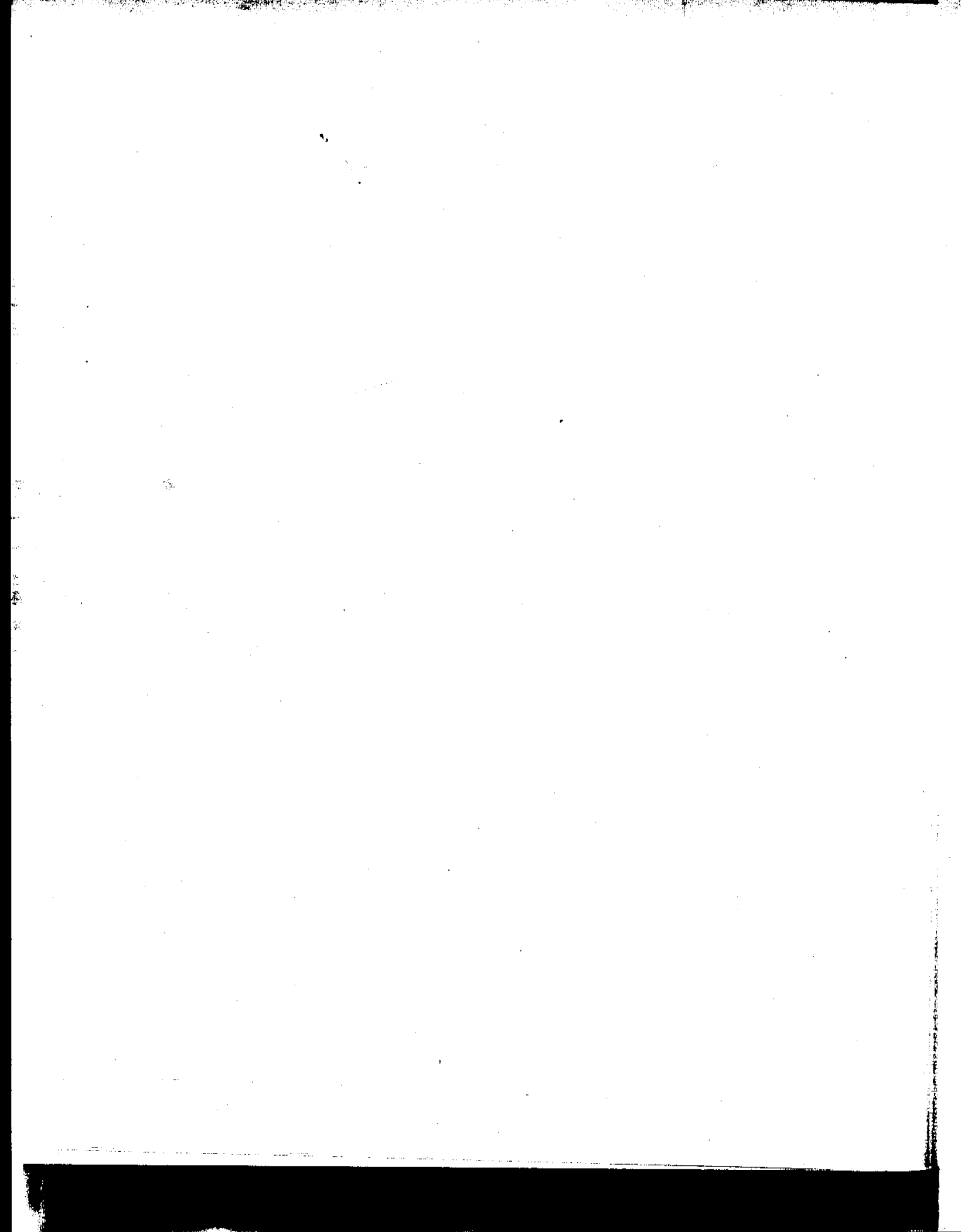
system: an aggregate of hardware and software organized to perform a function or functions.

tailing: a chromatographic peak which is skewed. Usually due to active sites in the column or the chromatographic system.

variability: used to describe a chromatogram with retention times and peak areas that are not reproducible.

vial: the 2 ml or 100 μ l bottles used for holding samples. See **bottle**.

viscosity: a flow characteristic of a liquid that may influence the reproducibility of the injection volume.



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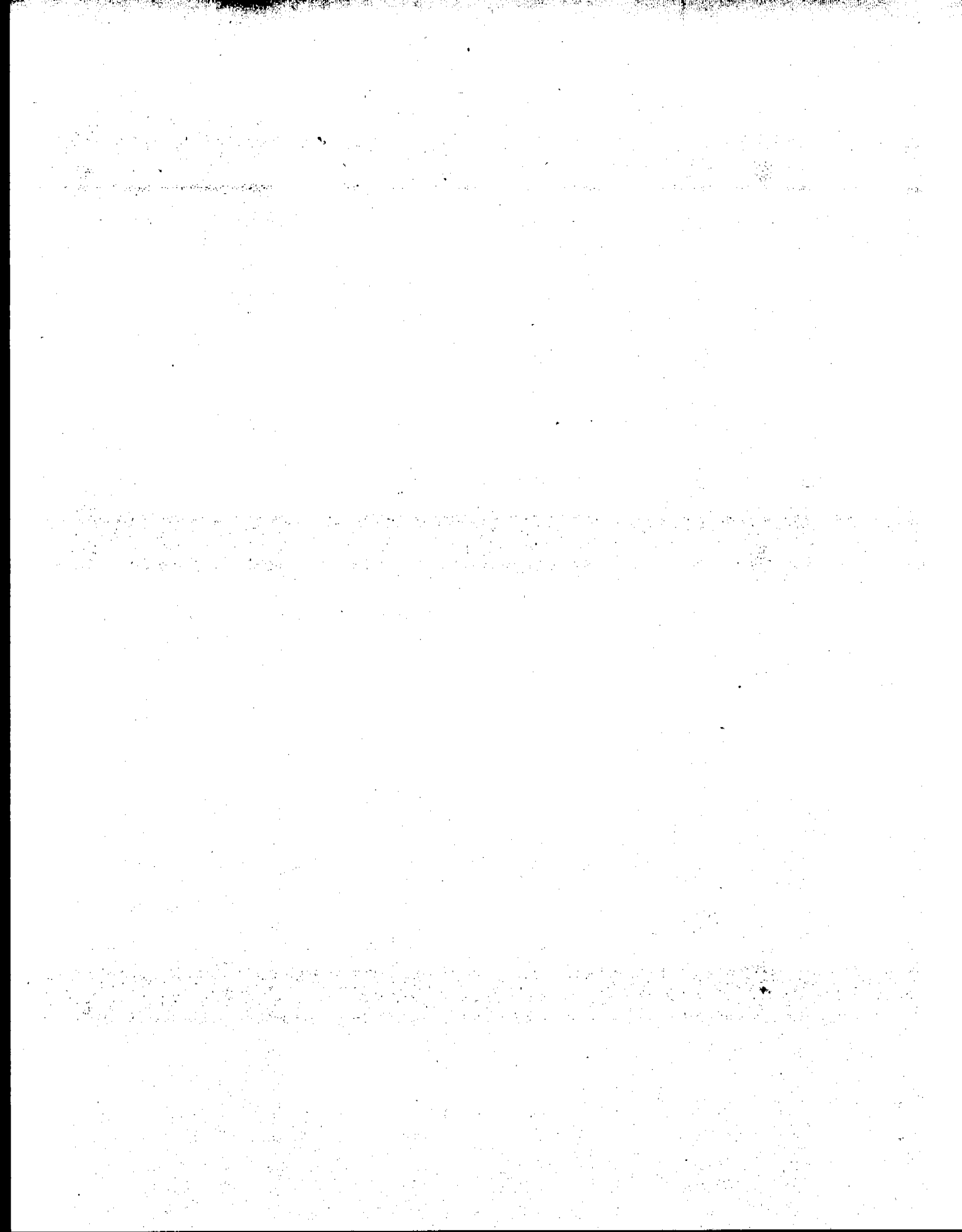
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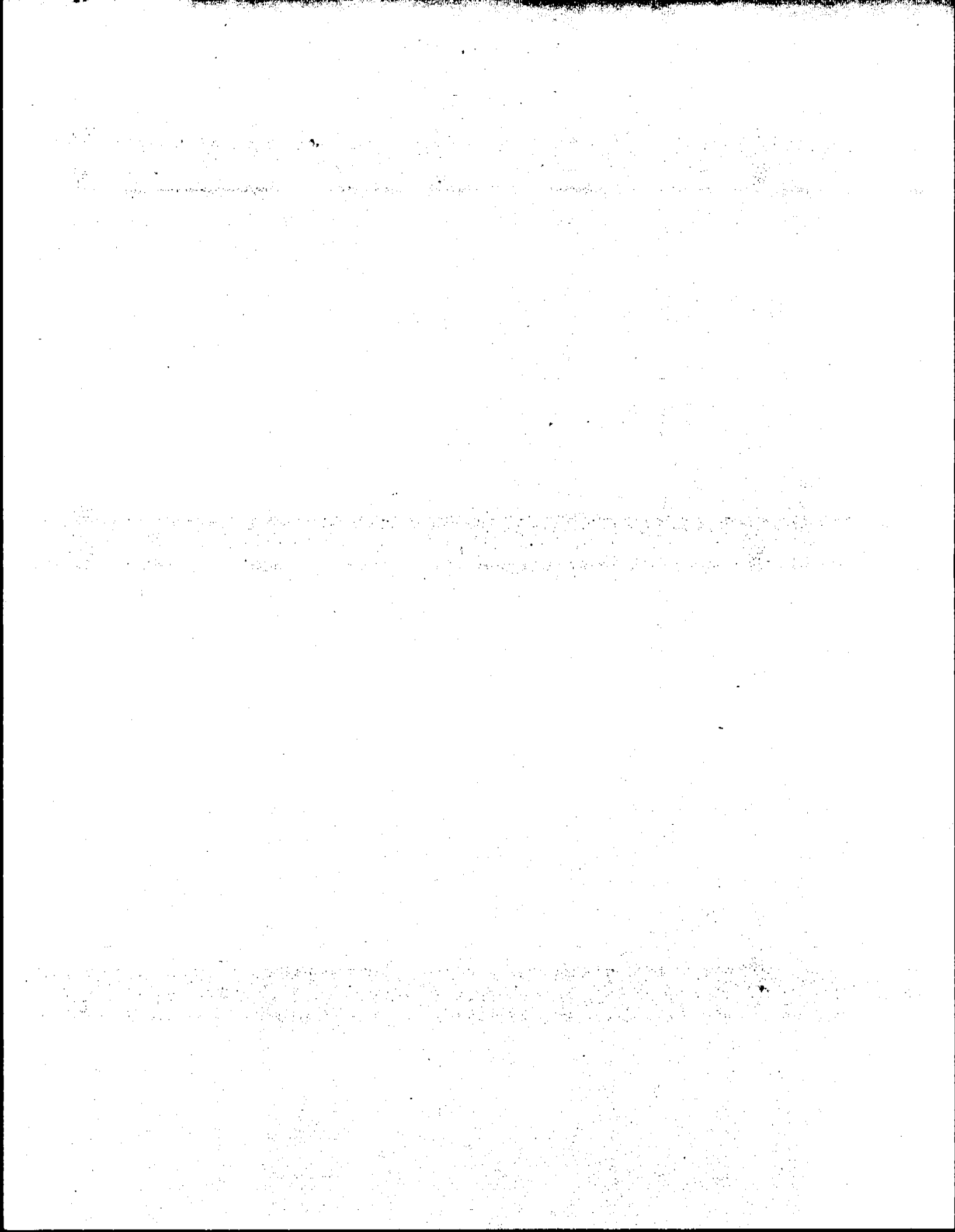
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