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# 507 AUTOSAMPLER

# INSTALLATION AND OPERATION MANUAL

**Part No. 015-240651**
**Rev. D**

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**BECKMAN**

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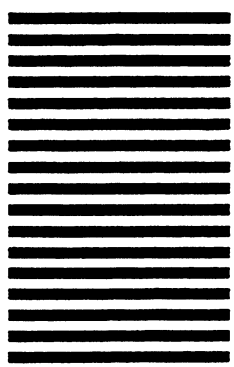
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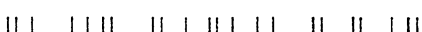


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# ⚠ SAFETY NOTICE

## INTRODUCTION

This page summarizes information basic to the safe operation of the modules and devices covered by this manual.

The international symbol ⚠ displayed on the modules and in this manual is a reminder to the user that all safety instructions should be read and understood before installation, maintenance and servicing of the modules are attempted.

When the symbol ⚠ is displayed in this manual, pay special attention to the specific safety information associated with the symbol.

## SAFETY DURING INSTALLATION

The HPLC and P/ACE systems covered in this manual are designed to be user installed and maintained. Installation, troubleshooting, and maintenance procedures for the modules and devices comprising the systems are covered in individually tabbed sections of this manual in addition to applicable safety precautions.

- Any servicing of this equipment which requires the removal of any covers or panels can expose parts which involve the risk of electric shock or personal injury. Refer such servicing to qualified personnel.

- When shipped from the factory, the modules and devices are equipped and adjusted for operation on a nominal 120V, 50-60 Hz power supply. Follow the procedures in the individual module section for conversion to other voltages.

- The systems described in this manual are designed for use with specific support or accessory equipment, e.g., personal computer, printer, etc., made by other manufacturers. This support equipment is described in the manual. The use of any equipment other than those intended for use by Beckman has not been evaluated for safety. Use of any accessory equipment not specifically recommended in this manual shall be the sole responsibility of the user.



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## ELECTRICAL SAFETY

To reduce risk of electrical shock, all module and devices employ a three wire electrical cable and plug to connect the equipment to earth ground. To preserve this safety feature:

- Make sure the matching wall outlet receptacle is properly wired and earth grounded.
- Never use a three-to-two wire isolating plug adapter.
- Never use a two wire extension cord or a two wire multiple-outlet power strip.

## SAFETY AGAINST RISK OF FIRE

- Certain electrical circuits within each module are protected by fuses against overcurrent conditions. For continued protection against the risk of fire, replace fuses only with the same type and rating specified.

## CHEMICAL AND BIOLOGICAL SAFETY

Normal operation and some maintenance procedures require the use of many reagents which may be toxic, flammable or biologically harmful.

- The user must observe all cautionary information printed on the original solution container(s) prior to their use.
- Infectious samples must be handled according to good laboratory practices and methods to prevent spread of disease.
- All waste solutions must be disposed of in a proper manner.
- When using nitric acid to clean the detector flow cell, observe standard laboratory safety practices. Wear protective eye covering and immediately wash any splashes from skin or clothing with plenty of water.



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## HIGH PRESSURE SAFETY

The HPLC Solvent Modules (Pumps) generate extreme high liquid pressures. Special alloy metals or other materials are used in all liquid handling components to resist chemical degradation and the pressures involved.

- All fittings and tubing connections must be made tight and secure enough to prevent leaks, but they must not be overstressed during tightening.
- Worn or damaged parts should be replaced with parts supplied by Beckman Instruments only.
- All leaks should be wiped up immediately and the leakage cause corrected.
- Disconnect the system from the electrical supply source before opening any high pressure connections. Loosen the connections slowly to allow any residual pressure to bleed down to a safe level before opening fully.
- Never attempt to block the open end of any pressurized tubing or liquid coupling with the fingers or hand, as liquid injection under the skin could occur.
- It is recommended that safety glasses be worn whenever working on the HPLC system.

## SOURCE LAMP SAFETY

The source lamp(s) used in the detector modules operate at high temperatures.

- To avoid a possible electric shock, disconnect the instrument from the electrical power source before servicing or or servicing the lamp(s).
- To prevent a possible burn, allow the lamp(s) to cool at least five minutes before handling.
- The deuterium (D2) lamp generates UV light. Do not look directly at an operating lamp. without wearing UV protective eye glasses.


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# Operating Instructions

This document is provided to help you establish safe conditions in operating your equipment. Specific considerations and precautions are also described in the manual, but appear in the form of **Warning**, **Caution**, and **Note.** It is important that you operate your equipment in accordance with this instruction manual and any additional information which may be provided by Beckman. Address any questions regarding the safe operation of your equipment to your nearest Beckman Sales Office or Service Center.

**⚠ WARNING**

**The word WARNING precedes important information identifying an imminently hazardous situation which, if not avoided, will result in death or serious injury.**

**⚠ CAUTION**

**The word CAUTION precedes information indicating a potentially hazardous situation which, if not avoided, may result in minor or moderate injury and/or damage to your equipment. It may also be used to alert against unsafe practices.**

**NOTE**

**The word NOTE precedes important information which should be followed during installation, use or servicing of equipment.**



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**Listed below are the WARNINGS and CAUTIONS appearing in 507 Autosampler Installation and Operation Manual**

**⚠ WARNING**

**To avoid personal injury, do not plug power cord into MAINS power panel when performing the removal and reinsertion of the Fuse/Line Voltage Selector Cartridge.**

**⚠ CAUTION**

**To avoid damage to the pneumatics in the instrument, clean, dry air or inert gas must be used.**

**⚠ CAUTION**

**Avoid overtightening as this may cause damage to the fitting.**

**⚠ CAUTION**

**Do not use glass inserts in the standard 2 mL vials. The inserts make the opening too small and can result in a bent needle if the needle hits the insert. The only recommended vial and insert combination are available from National Scientific Company (Norcross, GA; 1-800-332-3331), catalog item C4011-1 (vial), C4010-630 (insert), C4011-1A (crimptop seal).**

**⚠ CAUTION**

**Do not reuse a vial without replacing its septum as this may cause faulty sealing of vial.**

**⚠ CAUTION**

**To avoid damaging the needle, do not move the tray when the needle is down.**

**⚠ CAUTION**

**Never allow a buffer or salt solution to remain in the system during extended shutdown because crystalization can occur. If salt buffers have been in use, then flush with water, not IPA which may cause precipitation. Also, it is preferable that the counter block of the DDS is loosened by two turns counter-clockwise. Refer to Figure 4-1 for location of counter block.**



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**⚠ WARNING**

**When the needle shield is removed, the needle is exposed and personal injury may occur if an injection occurs while working in this area.**

**⚠ CAUTION**

**The lifetime of the pump tubing will be reduced significantly if the counterblock is adjusted such that the tubing is pressed very tight.**

**⚠ CAUTION**

**Over tightening of the nut into the injection valve may cause the ferrule to close off the tubing.**

**⚠ WARNING**

**Be sure to disconnect the air supply to prevent personal injury! Failure to disconnect the air supply may result in accidental puncturing of your finger while working with the needle assembly if the needle assembly is activated.**



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# WARRANTY

Subject to the exceptions and upon the conditions stated below, the Company warrants that the products sold under this sales agreement shall be free from defects in workmanship and materials for one year after delivery of the products to the original Purchaser by the Company and if any such product should prove to be defective within such one year period, the Company agrees, at its option, either (1) to correct by repair or at the Company's election by replacement, any such defective product provided that investigation and factory inspection discloses that such defect developed under normal and proper use, or (2) to refund the purchase price. The exceptions and conditions mentioned above are as follows:

(a) Components or accessories manufactured by the Company which by their nature are not intended to and will not function for one year are warranted only to give reasonable service for a reasonable time. What constitutes a reasonable time an a reasonable service shall be determined solely by the Company. A complete list of such components and accessories in maintained at the factory.

(b) The company makes no warranty with respect to components or accessories not manufactured by it. In the event of defect in any such component or accessory the Company will give reasonable assistance to Purchaser in obtaining from the respective manufacturer whatever adjustment is authorized by the manufacturer's own warranty.

(c) Any component claimed to be defective must, if required by the Company, be returned to the factory, transportation charges prepaid, and will be returned to Purchaser with transportation charges collect unless the product is found to be defective, in which case the Company will pay all transportation charges.

(d) If the product is a reagent or the like, it is warranted only to conform to the quantity and content and for the period (but not is excess of one year) stated on the label at the time of delivery.

(e) The Company may from time to time provide a special printed warranty with respect to a certain product and where applicable, such warranty shall be deemed incorporated herein by reference.

IT IS EXPRESSLY AGREED THAT THE ABOVE WARRANTY SHALL BE IN LIEU OF ALL WARRANTIES OF FITNESS AND OF THE WARRANTY OF MERCHANTABILITY AND THAT THE COMPANY SHALL HAVE NO LIABILITY FOR SPECIAL OR CONSEQUENTIAL DAMAGES OF ANY KIND OR FROM ANY CAUSE WHATSOEVER ARISING OUT OF THE MANUFACTURE, USE, SALE, HANDLING, REPAIR, MAINTENANCE, OR REPLACEMENT OF ANY OF THE PRODUCTS SOLD UNDER THIS SALES AGREEMENT.

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## MODEL 116 & 126 SEAL REPLACEMENT GUARANTEE:

A spare seal is included with the 116 and 126 Solvent Delivery Modules. When and if it is used, a replacement seal will be provided at no charge, upon request by the owner. Replacement seals will be sent by mail to the owner and the owner has the responsibility of installing the seals. Replacement pistons or other expendable parts are not covered under warranty. This guarantee is in effect for five years from delivery of the system. Seals in the 110B are not covered.

## BIOCOMPATIBILITY GUARANTEE:

All System Gold Chromatography Systems, including the Model 338 Gradient System, are covered by a special biocompatibility guarantee where the systems, when operated under conditions recommended by BII, are guaranteed to provide recovery of biological activity to a level meeting or exceeding that provided by comparable chromatography systems irrespective of the materials of construction. If such biological activity cannot be attained, then BII will at BII's option, repair or replace the system or accept return of the product and refund the original purchase price thereof. This guarantee is for a period of one year from date of delivery and applies to systems only, not to components, columns or other parts purchased individually.

## SYSTEM GOLD SOFTWARE UPDATE GUARANTEE:

Registered licensees of System Gold Chromatography Software packages are entitled to the following:

1. New versions of System Gold software which incorporate changes to the release purchased by the customer will be provided for a period of one year after delivery of the original software package.

2. Special prices on new releases of software which provide changes in functionality and/ or specification.

3. Disk replacement program which provides users with replacements for damaged disks for up to one year after delivery of the original disk(s) at a charge of $50.

For additional information, please refer to the Software License Agreement for Beckman System Gold Chromatography Software (ASW-2).



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## WARRANTY ON COMPONENTS MANUFACTURED FOR BECKMAN

Specific components manufactured for BII are warranted for one year from date of delivery. These items are as follows:

Model 502 Autosampler
Model 507 Autosampler
Model 231 Post Column Reactor
Model 235 Column Heater
Model 427 and 427M Integrator
Model BD-40 and 41 Chart Recorders
Model 156 Refractive Index Detector
Model 157 Fluorescence Detector

## WARRANTY ON DETECTOR LAMPS:

Lamps for Beckman manufactured detectors are warranted for 500 hours; for non-Beckman manufactured detectors, 500 hours or one year, whichever occurs first. All replacement lamps are customer installed.

## WARRANTY ON SYSTEM GOLD CHROMATOGRAPHY SOFTWARE:

Please refer to Software License Agreement for Beckman System Gold Chromatography Software (ASW-2).

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# Section 1
# Introduction

## 1.1 INTRODUCTION

The Beckman Model 507 Autosampler shown in Figure 1-1 is designed for unattended automatic injection of samples into an HPLC system. Setup and operation of the 507 Autosampler is made easy through the convenient location of the front panel controls and keyboard.

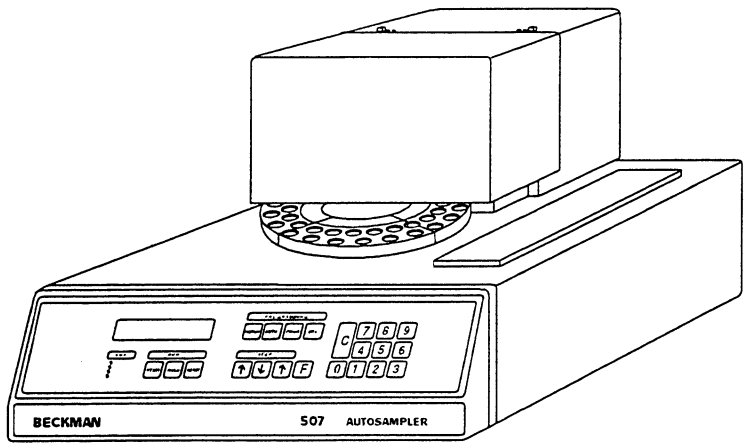



**Figure 1-1 Beckman 507 Autosampler**

The 507 Autosampler has the capability to be operated in the System Gold™ network. Once connected to the network, simply pressing the letter **F** followed by the number **4** will place the unit on-line. If the unit is to be operated off-line (stand-alone), first power down the unit. Next, turn the power back on. The unit will automatically come up in the keyboard (stand alone) mode. A flip up cover allows access to plumbing and pneumatics located on the upper front area of the unit.

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The 507 Autosampler is flexible in its sample handling, being capable of executing up to 99 consecutive runs, i.e., series, with each series comprising from 1 to 96 sample vials. Different programmable methods, i.e., inject volume, analysis time, etc., can be called for each series, thus allowing for individual handling of each sample vial.

Options are available to enhance performance of the 507 Autosampler and are discussed as follows:

- **Reagent Addition Cassette (RAC)**

  The customer installable RAC option permits on-line mixing of a reagent in a constant ratio with the sample. This option is useful for internal standard addition or for precolumn derivatization to save time and labor. This option will be referred to as RAC on the instrument Display where on power up, the STD (Standard) or RAC mode of operation is selected.

- **Sample Tray Cooling/Heating Unit**

  Customer installable, the Sample Tray Cooling/Heating Unit permits cooling labile samples down to 4 °C or warming hard-to-dissolve samples up to 60 °C. Cooling or heating is through the sample tray and temperature is controlled via an external circulating bath. The external bath is not included with this option.

- **Stream Switching Valves**

  For those applications requiring stream switching, factory installed high-pressure 6-port, 2-position valves are available. These stream switching valves permit such procedures as sample clean-up via precolumn, precolumn concentration, solid-phase extraction, or other uses of multiple columns such as heart cutting. The valves are controlled by the Events Method programming feature of the 507 Autosampler.

- **Column Oven**

  For those applications requiring increased column temperature stability and/or elevated temperatures for improved elution times. Temperature range is ambient (plus 5 °C) to 80 °C. The column oven can be programmed for an initial temperature and one additional temperature.

## 1.2 SPECIFICATIONS

Refer to Appendix A of this manual for equipment specifications.

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# Section 2
# Installation

## 2.1 GENERAL

This section outlines the procedures necessary to unpack and set up the 507 Autosampler for operation. Information on setting up the various options including installation of customer installable options is covered in the Appendix of this manual. Follow the paragraphs in this section in sequence to install your 507 Autosampler.

## 2.2 UNPACKING

Before unpacking the 507 Autosampler, make sure an area has been cleared at the installation site. Also, make sure power and clean dry air is accessible at the site.

Carefully unpack the instrument and place at installation site. Be sure to remove the ship kit from the shipping container. Inspect the instrument for damage and make sure all items listed in the instrument's ship kit list are found in the ship kit. Notify carrier immediately if anything is damaged and/or missing. Refer to Appendix D of this manual for a list of spare parts.

**NOTE**

**Before proceeding with installation, open the protective cover of the injection system and remove the transport block securing the needle arm. Refer to Figure 2-1.**



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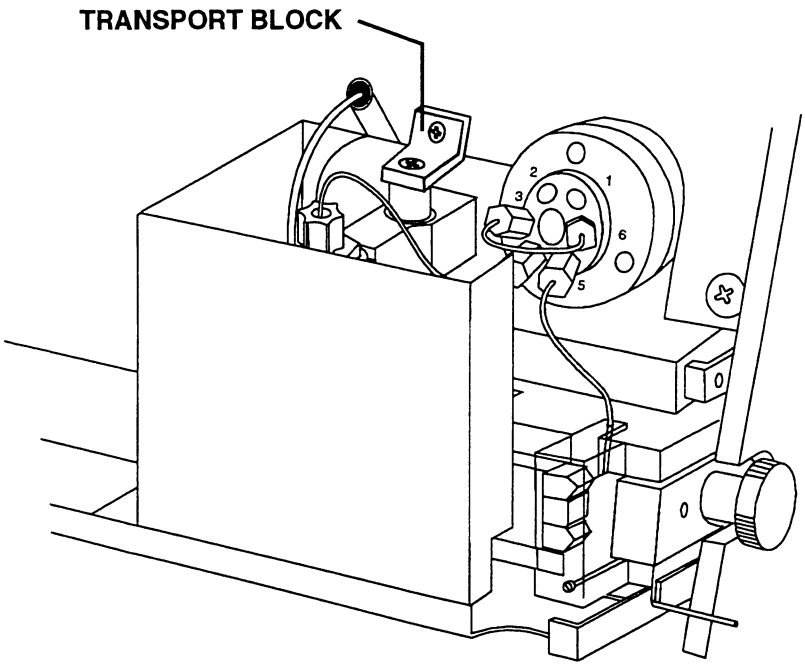



**Figure 2-1 Location of Transport Block**

**NOTE**

**At the installation site, be sure to allow sufficient room behind instrument to allow easy access to air, power, and communication connections.**



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## 2.3 POWER CONNECTIONS

Referring to Figure 2-2, proceed as follows:

**⚠ WARNING**

**To avoid personal injury, do not plug power cord into MAINS power panel when performing the removal and reinsertion of the Fuse/Line Voltage Selector Cartridge.**

1. Make sure MAINS power switch is turned off - "O" position on MAINS power panel. Note that power cord should not be plugged into MAINS power panel at this time.

2. Check voltage setting of instrument at the Fuse/Line Voltage Selector Cartridge on the rear panel. If indicated voltage is not correct:

   (a) Select proper voltage by removing, inverting, and then reinserting cartridge. **Make sure fuses are correct value (see 'b' below) before reinserting cartridge.** Correct orientation is when symbol on fuse holder lines up with on fuse panel as shown in Figure 2-2.

   (b) Replace fuses if necessary: For 100 to 120 Vac, use two 2 A 3AG Time-Lag fuses; for 220 to 240 Vac, use two 1 A 3AG Time-Lag fuses.

3. When you have determined that the voltage selection and fuses are correct for your power source, plug power cord into power receptacle on MAINS power panel and other end of power cord into AC outlet. Do not turn on power until called for.

## 2.4 COMMUNICATION CONNECTIONS

As shown in Figure 2-3, there are four terminal blocks located on the lower center area of the rear panel. These terminal blocks, CONTROL I/O, VIAL No OUTPUT, P3, and AUX, permit communication with external devices. All connections are made by insertion of a wire into the appropriate numbered pin and securing with a screw.

There are two connectors located on the lower left side of the rear panel. These connectors, CTRL OUT and CTRL IN are used only when the instrument is connected to the System Gold® environment.



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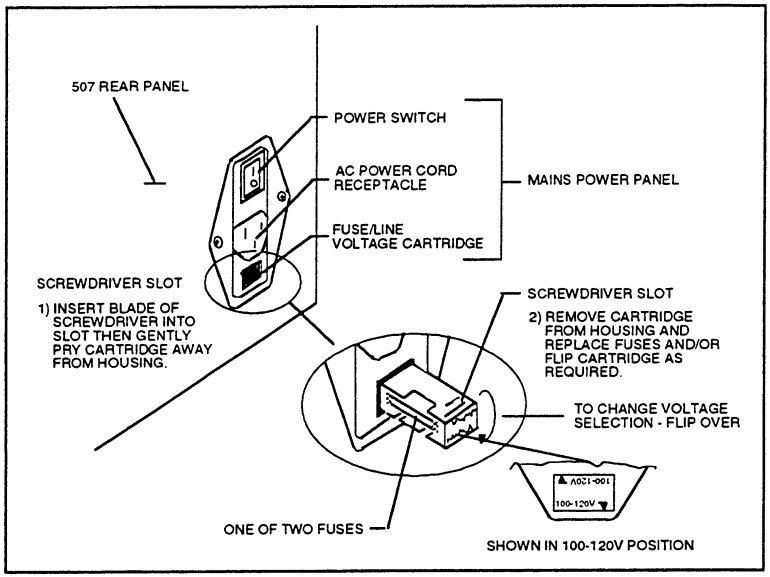



**Figure 2-2 MAINS Power Panel Showing Fuse/Line Voltage Selector Cartridge**

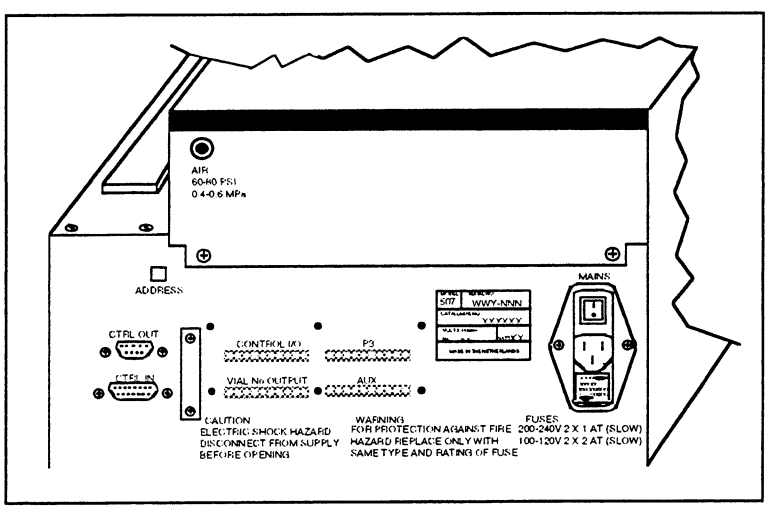



**Figure 2-3 Location of Communication Connections**

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The following paragraphs list the pin-out connections and functions for each connector. Use the following pin-out information to interconnect external devices to the 507 Autosampler.

### 2.4.1 CONTROL I/O Connector

The following list provides pin-out and functional description for the CONTROL I/O connector.

| Pin No. | Description |
|---|---|
| 1 | Injection Marker Output (active low, open collector, TTL). Use in conjunction with ground (pin 8). |
| 2/3 | Injection Marker Output (normally open contact). Contact is closed for 1.0 second starting at moment of injection. Connection is between pins 2 and 3. Ground (pin 8) is not used. |
| | **Note: The Injection Marker (negative edge or closing moment) can be used as a strobe/clock signal for reading all the other outputs.** |
| 4 | Labeled Vial Output (active low, open collector, TTL). This output goes low when a "labeled" vial is processed. |
| 5 | Vial Marker Output (active low, open collector, TTL). This output is low for 1.2 seconds starting 0.1 second before injection from a vial. |
| 6 | Stop I/O. Output goes low when instrument ends processing (READY on the display) and input forces instrument into RESET mode during analysis time (if it is pulled low). |
| 7 | Next Injection Input (negative edge, pulled up, TTL). This input triggers a next injection when instrument is in REMOTE CONTROL Mode. Also, Next Injection Input can be used as START INPUT when instrument is not in REMOTE CONTROL Mode. |
| 8 | Ground |

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| Pin No. | Description |
|---|---|
| 9 | Next Vial Input (negative edge, pulled up, TTL). This input triggers the next vial to be processed if instrument is in REMOTE CONTROL Mode. |
| 10 | Freeze Input (active low, pulled up, TTL). This input holds analysis time during the low period of this input. Use for external control of next injection by another device by setting this pin to ground. |

### 2.4.2 VIAL No OUTPUT Connector

The following list provides pin-out and functional description for the VIAL No OUTPUT connector. Eight-bit BCD Vial Number Output (true logic) is used, representing vial numbers 1-97. Refer to diagram at end of this paragraph for BCD information. Vial number 97 is the satellite position. Output is active when corresponding vial is processed.

| Pin No. | Description |
|---|---|
| 1 | Bit 0: Units |
| 2 | Bit 1: Units |
| 3 | Bit 2: Units |
| 4 | Bit 3: Units |
| 5 | Bit 0: Units x 10 |
| 6 | Bit 1: Units x 10 |
| 7 | Bit 2: Units x 10 |
| 8 | Bit 3: Units x 10 |
| 9 | Not Connected |
| 10 | Not Connected |

| Bit | 80 | 40 | 20 | 10 | 8 | 4 | 2 | 1 |
|---|---|---|---|---|---|---|---|---|
| BCD | 1 | 0 | 0 | 1 | 0 | 1 | 1 | 0 |
| | 8 | 7 | 6 | 5 | 4 | 3 | 2 | 1 |



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### 2.4.3 P3 Connector

The following list provides pin-out and functional description for the P3 connector. Four-bit BCD Solvent Selector Valve position output (true logic) for pins 2, 3, 4, and 5 is used. These functions can be programmed to control external devices.

| Pin No. | Description |
|---|---|
| 1 | +5 Volts, 500 mA max. |
| 2 | Bit 0 |
| 3 | Bit 2 |
| 4 | Bit 3 |
| 5 | Bit 1 |
| 6 | Ground |
| 7 | +24 Volts, 2 A max. |
| 8 | Not Connected |
| 9 | Not Connected |
| 10 | Not Connected |

### 2.4.4 AUX Connector

The following list provides pin-out and functional description for the AUX connector.

| Pin No. | Description |
|---|---|
| 1 | Not Connected |
| 2 | Not Connected |
| 3 | +24 Volts |
| 4 | Auxiliary Output No. 1 (active low, transistor). |
| 5 | Auxiliary Output No. 1 Inverted (active high,transistor). |
| 6 | Auxiliary Output No. 2 (active high, transistor). |
| 7 | Auxiliary Output No. 2 Inverted (active high, transistor). |
| 8 | Auxiliary Output No. 3 (active low, transistor). |
| 9 | Auxiliary Output No. 4 (active low, transistor). |
| 10 | Ground |

### 2.4.5 Module Wiring - HPLC System

Using the 507 Autosampler with an HPLC System requires specific wiring arrangements. Communications between the HPLC System and the 507 is through the CONTROL I/O terminal strip on rear panel. Several examples of wiring arrangements are shown in Figures 2-4 and 2-5.



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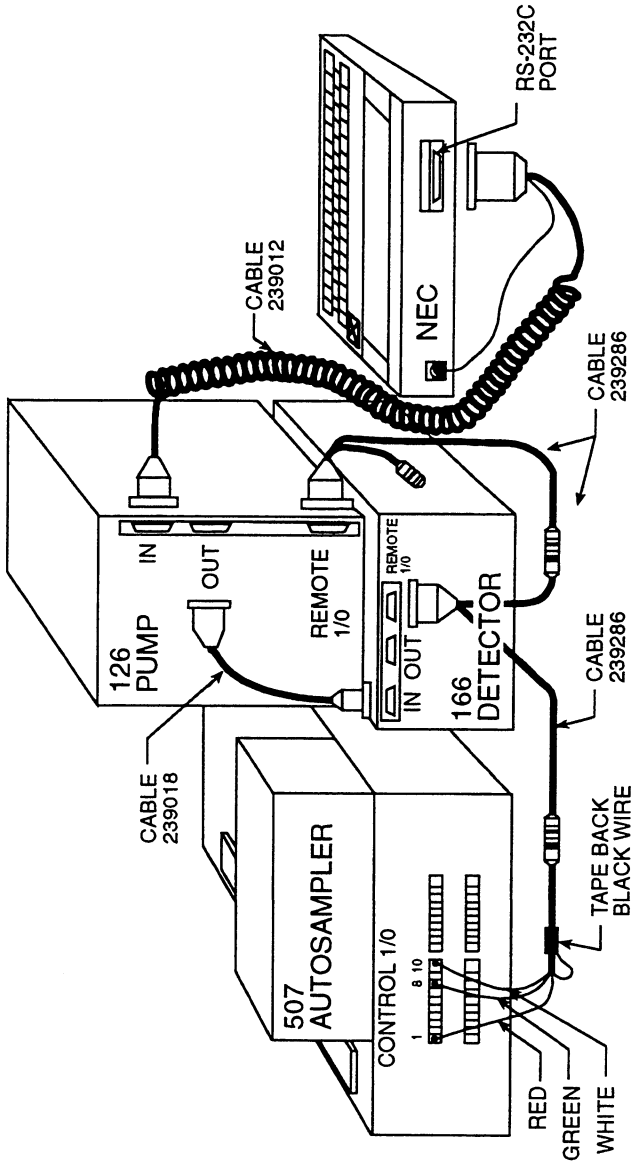



**Figure 2-4 Wiring Arrangement - NEC/507/126/166**



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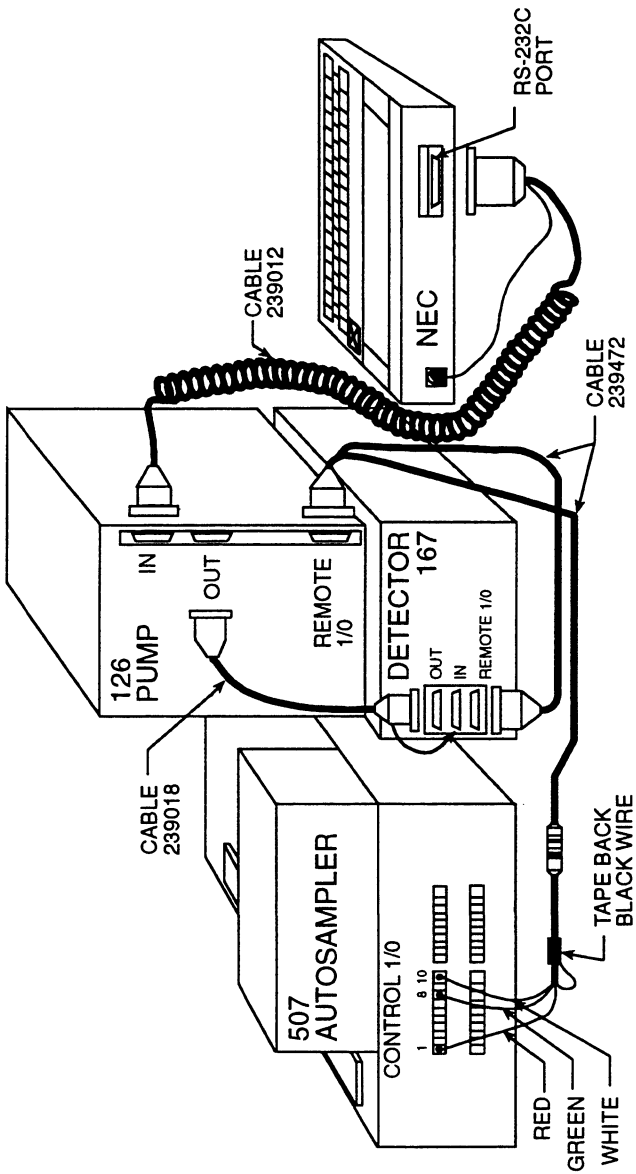



**Figure 2-5 Wiring Arrangement - NEC/507/167/126**



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### 2.4.6 Wiring 507 Output To 427 Integrator

Wire 507 Autosampler for starting the 427, then the Vial No. Output as shown below. Then, enter the commands in the 427 as outlined following the wiring information.

*Wiring Start Connections*

| 507<br>Control I/O | 427<br>External I/O |
|---|---|
| Pin 8 | B1 |
| Pin 1 | B5 |
| Pin1* | B14 |

*Only necessary if channel B is used.

*Wiring Vial No. OUTPUT Connections*

| 507<br>Vial No OUTPUT | 427<br>External Controls Option |
|---|---|
| Pin 1 | B6 |
| Pin 2 | A7 |
| Pin 3 | B7 |
| Pin 4 | A8 |
| Pin 5 | B8 |
| Pin 6 | A9 |
| Pin 7 | B9 |
| Pin 8 | A10 |

Enter the following commands in the 427:

1. Enter XD = 1 (enter out of Dialog Mode).

2. Enter the line: TT = 0.01 TF = "XD" TV = 1 (enter in Time Function Table of Dialog Mode).

   The vial number will now be printed in the report.



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## 2.5 AIR CONNECTION

A clean, dry source of air or inert gas is required to operate the pneumatics in the instrument. Using the filter/tubing assembly provided in the ship kit, connect push-in tube connector end into AIR fitting on upper left area of rear panel. Other end of assembly connects to the pressurized source of gas. Pressure should be adjusted to 0.4 to 0.6 MPa (60 to 80 psi). To disconnect connection at AIR fitting, push in rim (washer-shaped device at end of tubing) and simultaneously pull out tubing. Filter/tubing assembly is shown in Figure 2-6.

**⚠ CAUTION**

**To avoid damage to the pneumatics in the instrument, clean, dry air or inert gas must be used.**

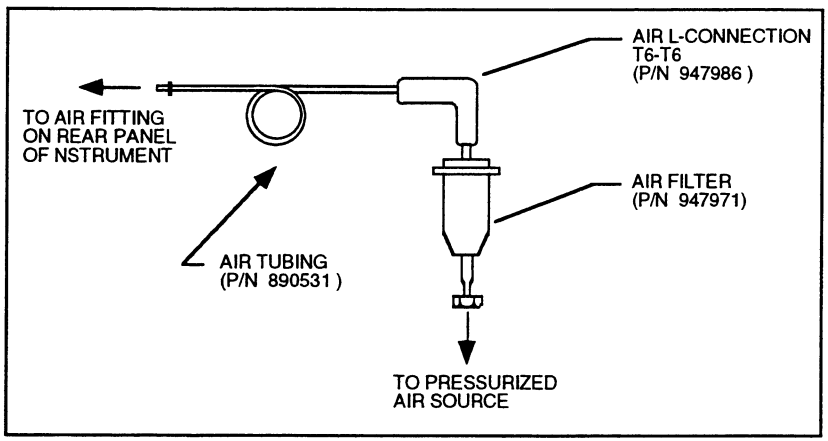



**Figure 2-6 Filter/Tubing Assembly**

## 2.6 PUMP/COLUMN CONNECTIONS

Before connecting the column and pump to the injection valve, make sure the HPLC pump and detector are installed to the right of the 507 Autosampler. Also note that this instrument has been tested with IPA (isopropanol). Make sure that the mobile phase of your HPLC system is miscible with IPA. If not, start up the system with an intermediate solvent as a mobile phase. Column should be disconnected during this intermediate step.



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Referring to Figure 2-7, make plumbing connections to the injection valve as follows:

1. Expose injection valve by lifting up the hinged cover.

2. Route high-pressure tubing (P/N 890948) from HPLC pump to port #1 of the injection (Rheodyne) valve. Note that the ship kit contains 2 kinds of Rheodyne nuts. Use the longer nuts for connections at the valve; they are easier to tighten/loosen with a wrench.

   Tighten fitting just enough to prevent leaks. You can check for leaks when the equipment is placed into operation.

**⚠ CAUTION**

**Avoid overtightening as this may cause damage to the fitting.**

3. Using the high-pressure tubing (890624) included in the ship kit, connect one end to port #2 of the injection valve. **Again use caution in tightening the fitting.**

**NOTE**

**Do not exchange column and pump connections at injection valve as this will prevent the contents of the sample loop from being injected in back flush onto the column. It is essential for proper operation that the contents of the sample loop be injected in back flush onto the column.**

4. Route remaining end of tubing out right side of instrument and connect to column inlet. As shown in Figure 2-7, the column compartment located on the top right area of the instrument cabinet provides a convenient location for placing the column. If the column heater option is installed, this compartment will contain the heater block.

5. Complete column installation by connecting appropriate tubing to column outlet and routing this tubing off to the right side of the instrument and to the detector.



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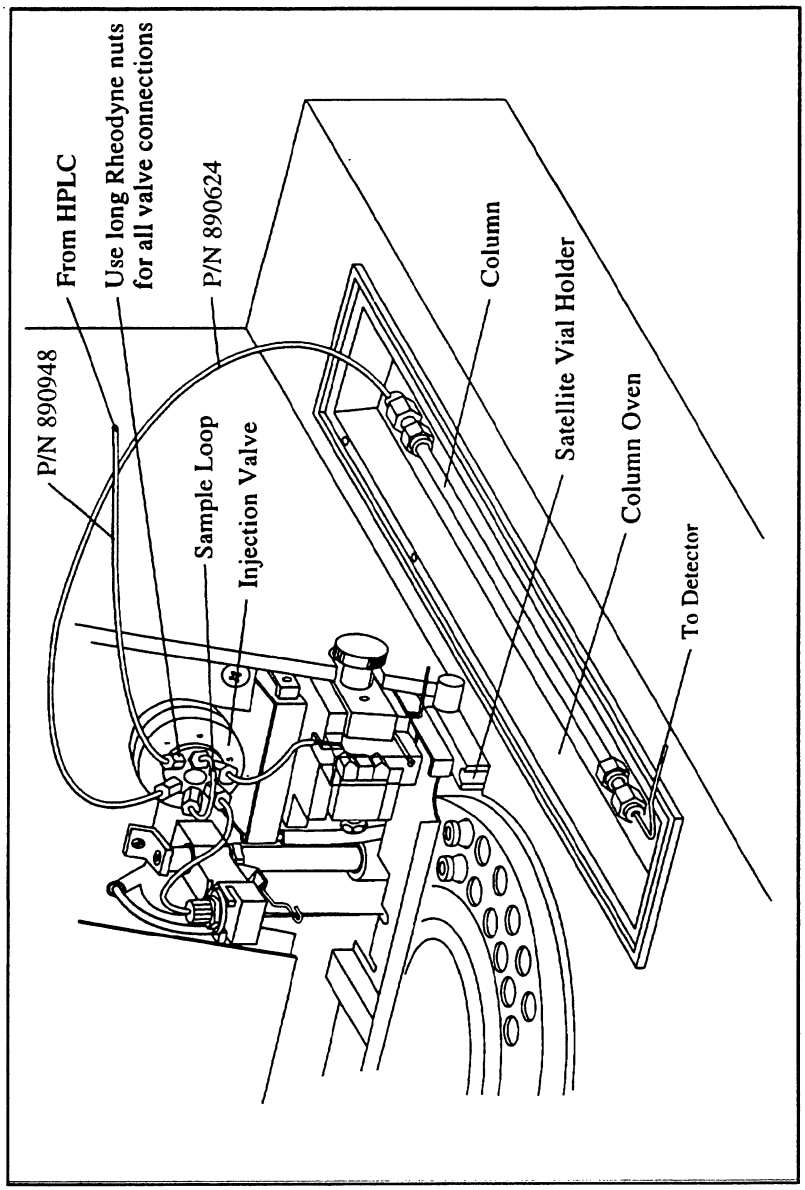



**Figure 2-7 Plumbing and Connections For HPLC Pump and Column**



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## 2.7 SAMPLE LOOP

The 507 Autosampler is shipped with a 100 µL sample loop installed. This loop allows for variable volume injection (partial loop filling) up to 60 µL, i.e., up to 60% of loop volume, or full loop injections of 100 µL. µL pickup volumes up to 40 µL may be used. If larger volumes are to be injected, the sample loop must be replaced.

For variable injection, the loop volume should be greater than 1.67 times maximum injection volume with a maximum of up to 10 times the injection volume. This is necessary to minimize the backflow of sample into the sample loop when switching from inject position (loop at system pressure) to load position (loop at atmospheric pressure).

For flushed loop injection, the loop volume should equal injection volume. Note that 1.5 times loop volume is withdrawn from sample vial as injection volume to fill loop quantitatively.

For µL pickup injections, the loop voluume should be:
2 (injection volume + 10 µL).


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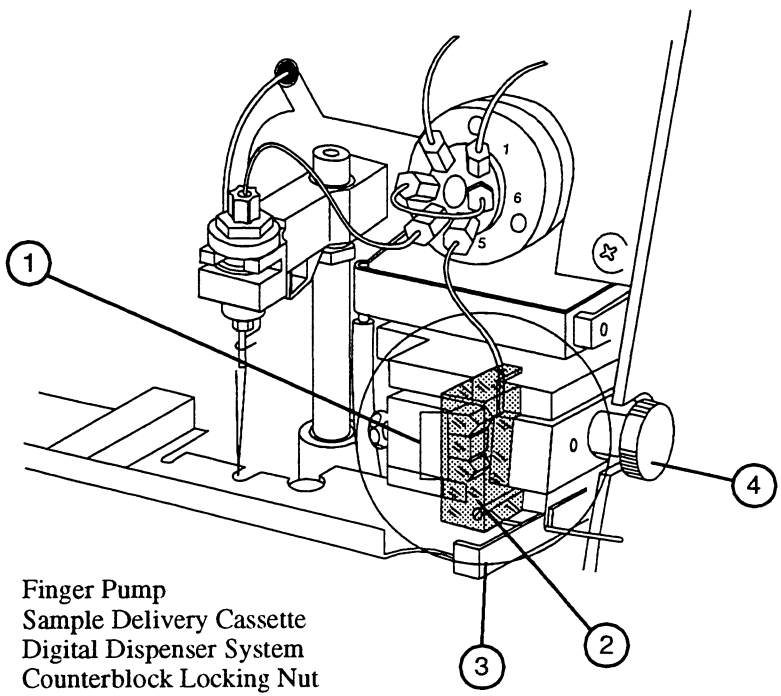
## 2.8 DIGITAL DISPENSER SYSTEM (DDS)

The Digital Dispenser System (DDS) is factory installed and connected to the injection valve. At the center of the DDS is the Finger Pump. This pump permits precise control of the volume of sample pulled into the sample loop. Each stroke of the pump is equivalent to 1 µL. If the DDS components require adjustment and/or maintenance, refer to Section 5, paragraph 5.2 of this manual. Refer to Figure 2-8 for location of DDS.

The 507 is shipped with the DDS's Counterblock Locking Nut loosened. Before beginning to use the autosampler, gently tighten this locking nut with your fingers.

**NOTE**

**Whenever theautosampler is not to be used, loosen the Counterblock Locking Nut to prevent the DDS tubing from developing a memory. If you do not do this, it will be necessary to run the DDS for 800 cycles before beginning automated operation.**




1. Finger Pump
2. Sample Delivery Cassette
3. Digital Dispenser System
4. Counterblock Locking Nut

**Figure 2-8 Digital Dispenser System (DDS)**



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## 2.9 VIAL PREPARATION

The following paragraphs cover general information on vial size, consumption, filling, and loading/positioning on sample trays.

### 2.9.1 Vial Requirements

The standard sample vials included in the ship kit are 2 mL with screw cap (P/N 887954). These vials can also be ordered from Sun-Brokers under catalog number 1260A.

For small sample volumes or for low sample loss (low rest volume), use Chromacol 200 or 300 µL conical vials.

Where needle wash is required, a satellite vial can be used. The standard satellite wash vial is 4 mL. The vial kit is available from Beckman under P/N 240649 or from Wheaton under P/N 224822.

**⚠ CAUTION**

**Do not use glass inserts in the standard 2 mL vials. The inserts make the opening too small and can result in a bent needle if the needle hits the insert. The only recommended vial and insert combination are available from National Scientific Company (Norcross, GA; 1-800-332-3331), catalog item C4011-1 (vial), C4010-630 (insert), C4011-1A (crimptop seal).**

### 2.9.2 Sample Usage

There are three factors that affect sample usage, i.e., rest volume, flush volume, and injection volume. Each factor is discussed in the following paragraphs.

*REST VOLUME*

Since the sample needle is always at some (very short) distance above the bottom of the sample vial, a certain volume of sample always remains in the vial, out of reach of the sample needle. This volume is called the rest volume. For standard 2 mL vials, the rest volume is 200 µL. For a conical vial, the rest volume is only a few microliters.



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### *FLUSH VOLUME*

Prior to the first injection from a sample vial, the sample needle and tubing (needle —> injection valve) must be quantitatively filled with sample. Depending on the concentration differences between samples and the cross-contamination allowed, this flush volume ranges between 30-99 µL (programmable). Subsequent injections from the same vial are preflushed with half the specified flush volume. Refer to Table 2-1 for more information on flush volume in reference to injection mode.

### *INJECTION VOLUME*

Refer to Table 2-1 for details on injection volume as related to injection mode, flush volume, and wash volume.

| | | | Loop Fill Volume | |
|---|---|---|---|---|
| Full Loop | Flush Volume | 1/2 Flush Volume | 150% Loop Volume | Wash as specified by internal valve and method 99 |
| Partial Loop | Flush Volume | 1/2 Flush Volume | Specified Injection Volume (max 60% loop volume) | Wash as specified by internal valve and method 99 |
| µL Pick up | None | None | Specified Injection Volume (max 50% loop volume -10 µL) | 30 µL used to transport sample into loop |
| | *VOLUMES REMOVED FROM SAMPLE VIAL* | | | *VOLUMES FROM WASH VIAL* |

**Table 2-1 Injection Volume as Related to Injection Mode**


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Example:

Three regular injections from a standard sample vial with injection volumes of 20, 50, 100 µL, with a 100 µL loop, and a 30 µL flush volume, requires a minimum sample volume as calculated below:

$$200 + 30 + 20 + 0.5(30) + 50 + 0.5(30) + 1.5(100) = 480\ \mu L$$

### 2.9.3 Filling/Sealing Sample Vials

The 2 ml. vials, as well as the conical vials, can best be filled by means of a narrow-end pipette to allow air to escape when filling the vial.

Do not fill vials completely to avoid sample being forced into the air needle when the needle pair punctures the septum, resulting in cross-contamination of samples.

It is essential that the seal be airtight to maintain a constant pressure on the vial, especially with partial loop and µL pickup modes. Leaking vial septa will impair reproducibility. For screw cap vials, tighten the caps finger tight. If using crimp cap vials, check seal after crimping. If cap can be turned easily, the seal is not airtight. Readjust handcrimper.

**⚠ CAUTION**

**Do not reuse a vial without replacing its septum as this may cause faulty sealing of vial.**

### 2.9.4 Loading The Sample Tray

The sample tray consists of four removable segments that fit only in one position on the tray transport mechanism. The tray can be rotated manually, using the grip in the center of the tray, to move the segments to more accessible positions.

**⚠ CAUTION**

**To avoid damaging the needle, do not move the tray when the needle is down.**



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### 2.9.5 Positioning Of Vials

Sample vials or a series of sample vials may be placed in any position(s) on the tray. Vials of one series (same method of analysis for all vials of a series) should be placed in sequential positions on the tray. Empty spaces between the first and last vials of a series are allowed, since the instrument will detect empty positions and rotate the tray until the next vial of the series arrives at the injection position. If additional samples are to be expected for a certain series during the processing of that series, empty positions can be deliberately included in a sample series program.

Since the system (typically using the NEC controller in the Beckman HPLC system) and 507 Autosampler are programmed independently, it is necessary to match the number of vials and injections between the system and 507.



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# Section 3
# Operation

## 3.1 GENERAL INFORMATION

This section of the manual is arranged to lead you step-by-step through all aspects of operating the instrument. You will start off with a basic functional description of the keyboard and display followed by detailed procedures on programming the instrument. This section then concludes by utilizing the programming procedures described to perform automatic sample processing.

## 3.2 KEYBOARD DESCRIPTION

As shown in Figure 3-1, the keyboard is divided into three sections: **Program**, **Run**, and **Display**.

- The **Program** section contains the keys used to select a programming mode and to enter values for programmable parameters.
- The **Run** section contains the keys used to control the instrument when it is executing a program and shows the status of the auxiliary outputs.
- The **Display** shows the actual parameter and its value during programming or processing.

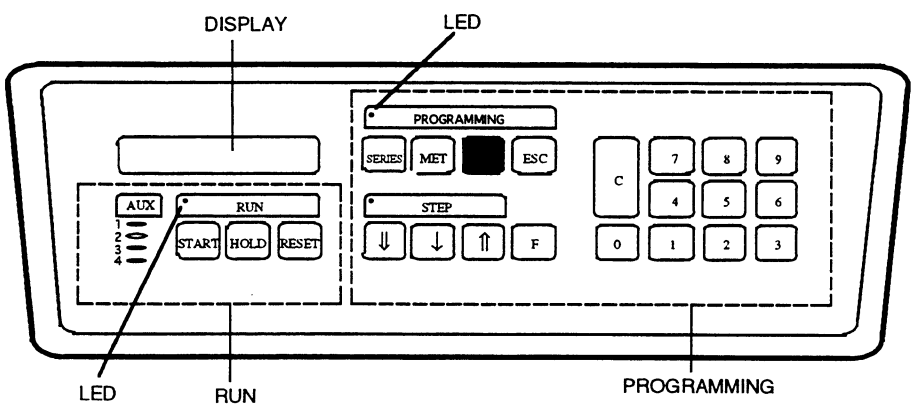



**Figure 3-1 507 Autosampler Keyboard**



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### 3.2.1 Keyboard Program Description

PROGRAMMING LED:
Indicates when in programming mode.

SERIES Key:
Selects Series programming mode.

METHOD Key:
Selects Method programming mode.

PRIORITY Key:
Selects Priority programming mode.

ESC Key:
Ends programming mode.

⇓ Key:
Permits stepping forward through main parameter list.

⇑ Key:
Permits stepping backwards through main parameter list.

↓ Key:
Permits stepping forward (substeps) through parameter sublist. Functions only when first character of lower line in the Display shows a numerical value.

C (Clear) Key:
Clears entered numerical values.

0 - 9 Keys:
Numeric keys used to enter value(s) for parameters shown on the Display.

F (Function) Key:
Function key used in combination with numeric key(s) to select special functions.



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### 3.2.2 Keyboard Run Description

START Key:
Starts execution of Series program

HOLD Key:
Freezes execution of RUN program during analysis time of sample being processed.

RESET Key:
Stops execution of RUN program and returns instrument to Ready status as shown on Display.

RESET LED:
Indicates error when flashing.

AUX LEDs :
Indicates status of auxiliary outputs 1 through 4.

### 3.2.3 Keyboard Display Description

The Display is a two-line LCD screen which provides visual information to make the operator aware of actual, special or error conditions of the instrument, data entries made when programming as well as other information requiring display. The information appearing on the Display will be referred to as appropriate throughout this manual.

## 3.3 PROGRAMMING

There are three catagories of programming: METH (Method), SERIES, and PRIOR (Priority). Each area of programming provides specific capabilities. Figure 3-2 illustrates these programming capabilities.

Although not a program as are METH and SERIES, RUN represents a combination of the METH and SERIES programs and also provides the means to coordinate the start of an analysis run incorporating the METH and SERIES programs.

Note that although you have not yet been instructed to turn on power, you can at this time. This will allow you to familiarize yourself with the following programming techniques before actually operating the instrument in an automatic mode.

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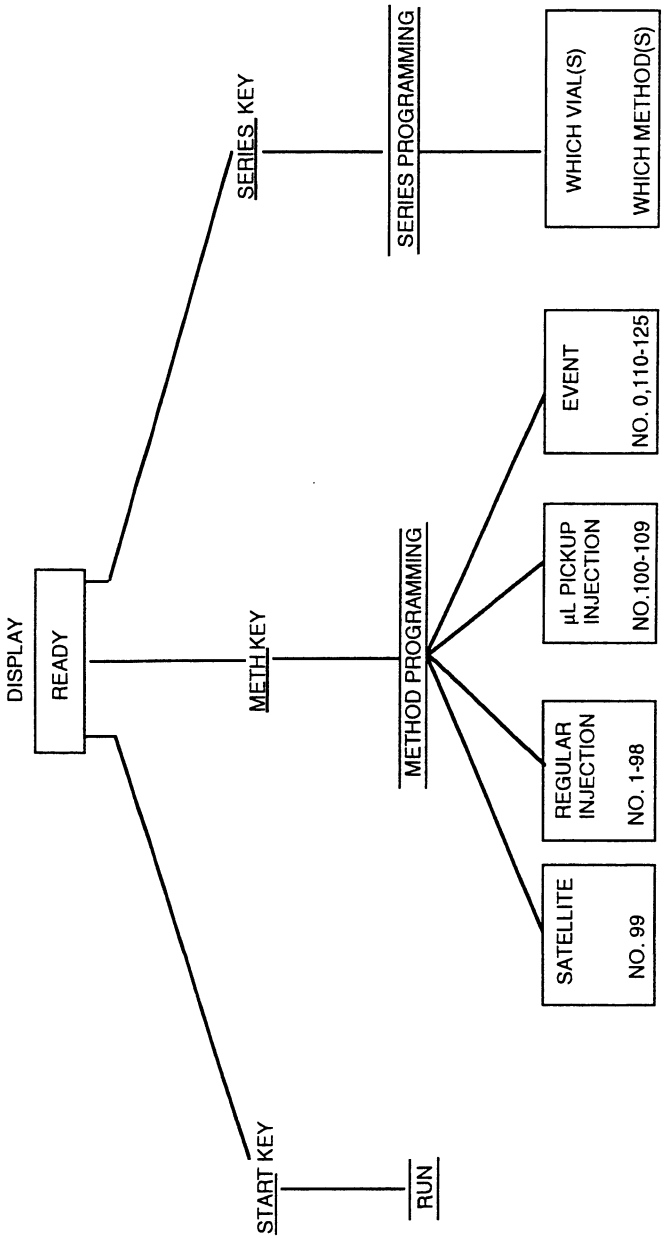



**Figure 3-2 Programming Chart**



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### 3.3.1 METH Programming

A METH program contains all instructions, e.g., analysis time, injection volume, auxiliary functions, etc. necessary to process a specific sample. One hundred twenty-five (125) programmed Methods can be saved in memory, each labelled with a METH file number. Methods may be programmed during RUN.

Methods that are already linked to certain samples, or series of samples, in one of the SERIES programs may be changed during RUN. However, the altered instructions are executed only in those SERIES programs that follow the SERIES program currently being executed.

There are four specific catagories or types of programs under METH. The specific program type is determined by a file number:

- **File Numbers 1 - 98.** These file numbers are designated *Regular Injection Methods.* Regular Injection Methods are used for flushed and partial loop fill injections.

- **File Number 99.** This file numbed is designated *Satellite Method.* The Satellite Method is used to process the vial at the off-tray position. The Satellite Method is linked to the Method via the Interval parameter which is addressed in the Regular Injection Method.

- **File Numbers 100 - 109.** These file numbers are designated for the *µL Pickup Methods.* These Methods includes a special injection routines for small sample volumes with zero sample loss.

**NOTE**

**The µL Pickup Method requires that the satellite vial be used to provide liquid to carry the sample into the loop.**

- **File Numbers 110 - 125.** These file numbers are designated *Event Method.* These Methods are used to control auxiliary outputs and stream switching valves. Event programs can be programmed on a time basis. Event Methods can be linked to other Methods by entering the appropriate Event Method File Number in the other Method program. Event Methods can also be linked directly to a SERIES program by entering the appropriate Event Method File Number in the SERIES program.



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Programming the instrument is accomplished via the keyboard. Keys are provided for stepping through program parameters and numeric keys for entering data (refer to paragraphs 3.3.1 through 3.3.3 for description of keyboard). Values are entered into memory when stepping to the next or previous program parameters. Programming parameters and data entries are shown on the Display.

Any valid keyboard action is confirmed by a short sound burst when pressing the key. Erroneous keyboard action is indicated by an intermittent sound burst.

The METH programming mode can be selected when instrument is in READY mode as shown on the Display or in RUN mode as indicated by the RUN LED.

**NOTE**

**If the instrument is in the SERIES programming mode as shown on the Display, press ESC key to call up the READY mode.**

Programming Tables 3-1 through 3-4 have been prepared for each METH programming technique. Refer to appropriate Table as listed on the following pages.

**Table 3-1 Regular Programming Methods**

**Table 3-2 Satellite Programming Methods**

**Table 3-3 µL Pickup Programming Methods**

**Table 3-4 Events Programming Methods**

Refer to appropriate Table as listed above, then proceed as outlined on the table.



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**Symbols:** [⇓]Next Step, [⇑]Previous Step, [↓]Substep

| [ ] KEY | DISPLAY | OPERATOR |
|---|---|---|
| | READY (X.X) STD | No action required. |
| [METH] | PROG. METH : 001 | Default value. Key in 1-98 to select a Regular Method. |
| [⇓] | PROG. METH : 001<br>TEMPERATURE 00 | Key in value of CCT column oven temperature (20-80°C). |
| [⇓] | PROG.<br>ANALYSIS TIME | Key in time. Maximum 99 min/59 sec. |
| [⇓] | PROG. METH : 001<br>EVENT METHOD 000 | Default value. Key in number of the Event Method to be used (110-125). If no events are to be used, enter 000. |
| [⇓] | PROG. METH : 001<br>LOOP VOLUME 000 | Default value. Key in volume of sample loop as installed. Maximum 999 µL. |
| [⇓] | PROG. METH : 001<br>1 INJECTION VOLUME: 000 | Key in volume for the first injection. |
| [↓] | PROG. METH : 001<br>2 INJECTION VOLUME: 000 | Key in volume for the second injection (from the same vial). |
| etc. | PROG. METH : 001<br>9 INJECTION VOLUME: 000 | Key in volume for the nineth injeciton (maximum). |
| [⇓] | PROG.<br>FLUSH VOLUME | Default value. Key in flush volume (30-99 µL). |
| [⇓] | PROG. METH : 001<br>INTERVAL VALUE 00 | Default value. Key in interval value "y". After every "y" vials, the satellite vial is processed according to Method 99. Check Method for appropriate program. |
| [ESC] | READY (X.X) STD | Instrument is in Ready status. |

**Table 3-1 Regular Programming Methods (1-98)**

**Symbols:** [⇓]**Next Step,** [⇑]**Previous Step,** [↓]**Substep**

| [ ] KEY | DISPLAY | OPERATOR |
|---|---|---|
| | READY (X.X) STD | No action required |
| [METH] | PROG. METH: 001 | Default value. Key in 99 to select a Satellite Method. |
| [⇓] | PROG. SATEL. METH: 099<br>TEMPERATURE 00 | Temperature and other parameters are Programmed as described for Regular Methods<br><br>INTERVAL PARAMETER, DES NOT OCCUR IN THE SATELLITE PROGRAM |
| [ESC] | READY (X.X) STD | Instrument is in Ready status |

**Table 3-2 Satellite Programming Methods (99)**



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**Symbols:** [⇓]**Next Step,** [⇑]**Previous Step,** [↓]**Substep**

| [ ] KEY | DISPLAY | OPERATOR |
|---|---|---|
| | READY (X.X) STD | No action required. |
| [METH] | PROG. METH : 001 | Default value. Key in 100-109 to select a µL. Pick Up Method. Key in 100 for example purposes. |
| [⇓] | PROG. PICKUP METH: 100<br>TEMPERATURE 00 | Key in value of CCT column oven temperature (20-80°C). |
| [⇓] | PROG. PICKUP METH: 100<br>ANALYSIS TIME 00:00 | Key in Analysis Time. Maximum 99 min/59 sec. |
| [⇓] | PROG. PICKUP METH: 100<br>EVENT METHOD 00:00 | Key in number of the Event Method to be used. If no events are to be used, enter 000. |
| [⇓] | PROG. PICKUP METH: 100<br>LOOP VOLUME 00:00 | Default value. Key in volume of sample loop as installed. Maximum 999 µL. |
| [⇓] | PROG. PICKUP METH: 100<br>1 INJECTION VOLUME 00:00 | Key in volume for the first injection. Must be <0.5 (loop volume) -10 µL. MAXIMUM OF 1 INJECTION/VIAL. |
| [ESC] | READY (X.X) STD | Instrument is in Ready Status. |

**Table 3-3 µL Pickup Programming Methods (100-109)**

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**Symbols:** [⇓]Next Step, [⇑]Previous Step, [↓]Substep

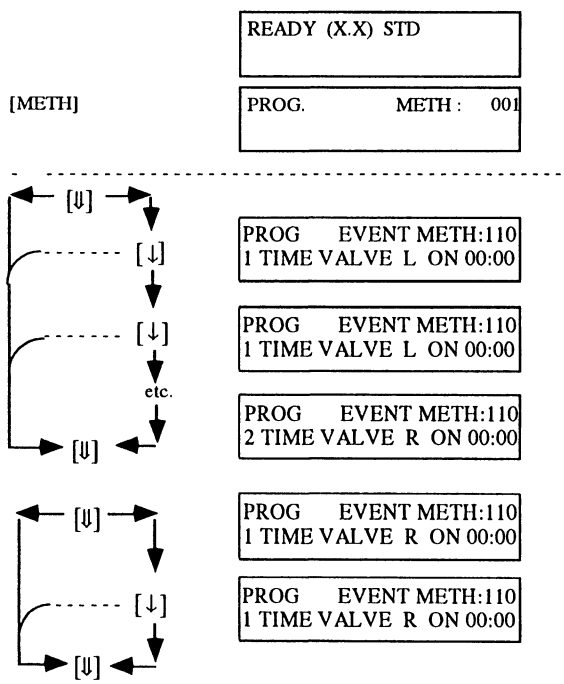



**Table 3-4 Events Programming Methods (110-125)**

**Symbols:** [⇓]Next Step, [⇑]Previous Step, [↓]Substep

| [ ] KEY | DISPLAY | OPERATOR |
|---|---|---|
| | | Instruments controlling external devices via the AUX Connector or rear panel of instrument. Refer to paragraph 2.4.4 for wiring details. |
| | | At each Event Method (110-125), up to 4 independent AUX outputs, i.e., AUX1, AUX2, etc. can be selected and individually turned on and off up to 4 times. The programmed time of each AUX output can overlap the selection of any or all of the other AUX outputs used in the Method. However, each AUX output can only be turned on and off sequentially, i.e., 1st time, 2nd, etc., up to the maximum 4 times. Each event can be programmed up to 99 minutes : 59 seconds. |
| [⇓] | | |
| [↓] | PROG. EVENT METH: 110<br>1 TIME AUX 1 ON: 00:00 | Key in first time (min/sec) for AUX 1 event to start. |
| [↓] | PROG. EVENT METH: 110<br>1 TIME AUX 1 OFF: 00:00 | Key in first time for AUX 1 event to stop. |
| [↓] | PROG. EVENT METH: 110<br>2 TIME AUX 1 ON: 00:00 | Key in second time for AUX 1 event to start. |
| [↓] | PROG. EVENT METH: 110<br>2 TIME AUX 1 OFF: 00:00 | Key in second time for AUX 1 event to stop. |
| etc. | | |
| [↓] | PROG. EVENT METH: 110<br>4 TIME AUX 1 OFF: 00:00 | Key in third and fourth AUX 1 event start and stop times as required. |
| [⇓] | | |

**Table 3-4 Events Programming Methods (110-125) continued**

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**Symbols:** [⇓]Next Step, [⇑]Previous Step, [↓]Substep

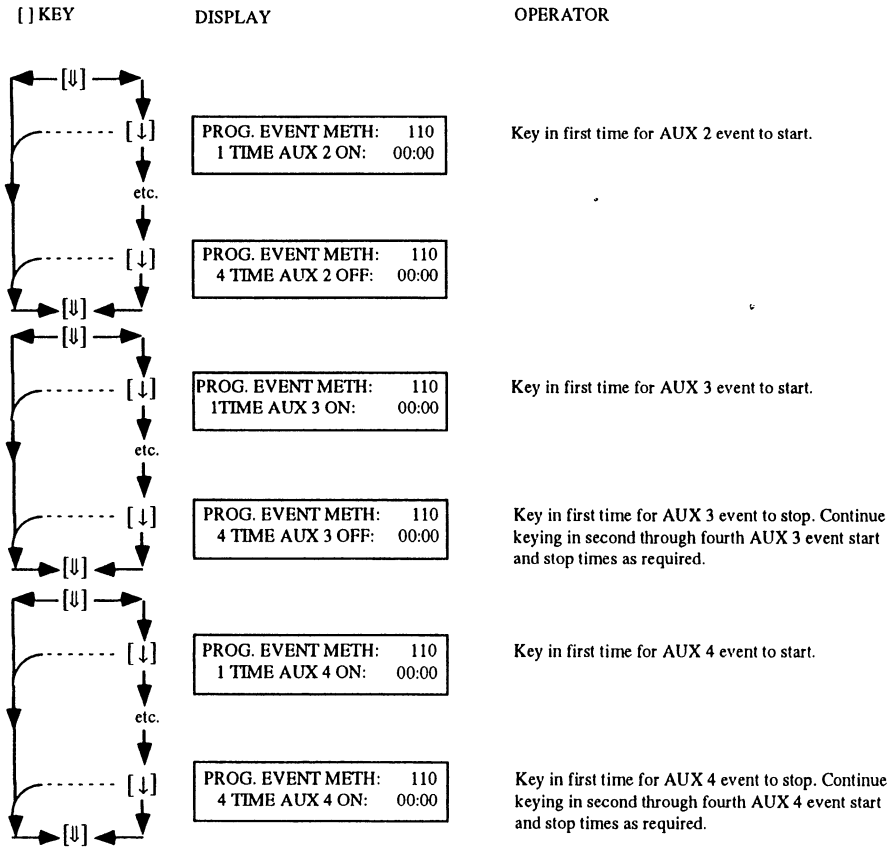



**Table 3-4 Events Programming Methods (110-125) continued**



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**Symbols:** [⇓]Next Step, [⇑]Previous Step, [↓]Substep

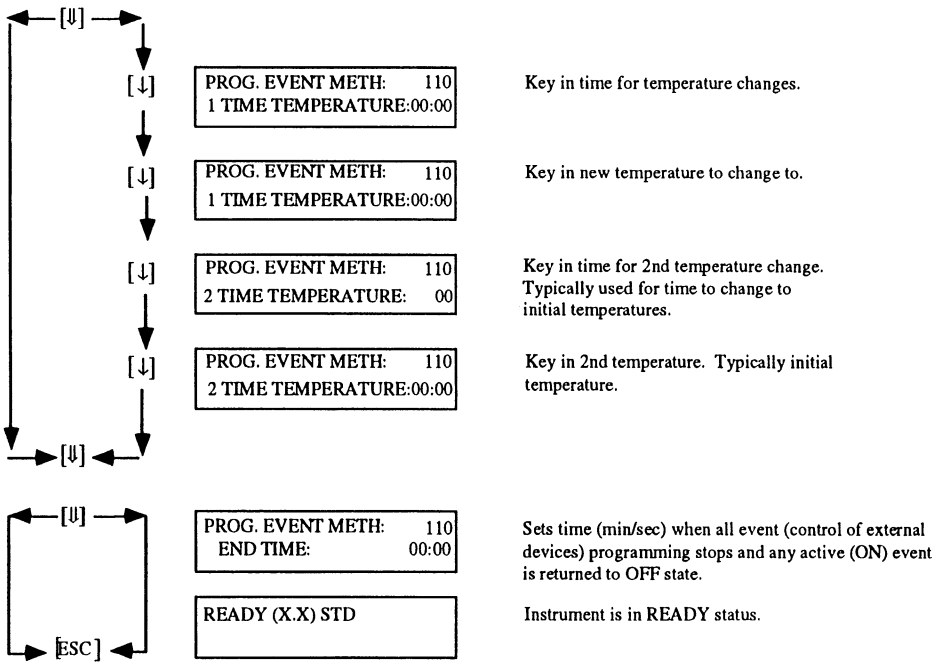



**Table 3-4 Events Programming Methods (110-125) continued**



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### 3.3.2 SERIES Programming

A SERIES program contains the information necessary to locate a sample series on the tray and determine which METH program will be used to process that series of samples.

SERIES programming permits execution of up to 99 sequential programs with each SERIES program comprising from 1 to 96 samples. The SERIES programs are executed sequentially during RUN. SERIES programs cannot be saved in RAM memory.

A SERIES program is created after the samples have been placed in the sample tray and after the Method(s) to process those samples have been programmed. A SERIES program will determine which samples are processed and how (determined by Method) they will be processed. A SERIES program can also contain instructions to change HPLC or other instrument parameters via AUX (auxiliary) outputs (EVENT Method is incorporated in the SERIES program) prior to the next sample series.

Programming the instrument is accomplished via the keyboard. Keys are provided for stepping through program parameters and numeric keys for entering data (refer to paragraphs 3.3.1 through 3.3.3 for description of keyboard). Values are entered into memory when stepping to the next or previous program parameters. Programming parameters and data entries are shown on the Display.

Any valid keyboard action is confirmed by a short sound burst when pressing the key. Erroneous keyboard action is indicated by an intermittent sound burst.

The SERIES programming mode can be selected when instrument is in READY mode as shown on the Display.

If instrument is in METH or PRIOR programming mode as indicated on the Display, press ESC key to call up the READY mode.

To create a SERIES program, refer to Table 3-5.

### 3.3.3 PRIOR (Priority) Programming

If a sample must be processed immediately, the RUN can be interrupted to allow for processing of the "priority sample". The vial position and Method to be used are programmable.



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**Symbols:** [⇓]**Next Step,** [⇑]**Previous Step,** [↓]**Substep**

| [ ] KEY | DISPLAY | OPERATOR |
|---|---|---|
| | READY (X.X) STD. | No action required. |
| [SERIES] | PROGRAMMING SERIES: 01 | Default value. Key in appropriate [Series] number. Press SERIES each time you want to advance to next series number, i.e.,02, 03, etc. |
| [⇓] | PROGRAMMING SERIES: 01<br>METHOD NUMBER 001 | Default value. Key in appropriate Method number. If the number of an Event Method is keyed in (110-125), no further parameters can be programmed; press [ESC]. |
| [⇓] | PROGRAMMING SERIES: 01<br>FIRST VIAL NUMBER 01 | Default value. Key in number of the first vial of the series of vials that should be processed according to Method previously selected. |
| [⇓] | PROGRAMMING SERIES: 01<br>LAST VIAL NUMBER 96 | Default value. Key in number of the last vial of the series of vials that should be processed according to Method previously selected. If the series of vials consists of only one vial; key in position of vial as first and last vial number. |
| [⇓] | PROGRAMMING SERIES: 01<br>1 LABLED VIAL 00 | Key in number of the first vial to which a Labled Vial signal output should be assigned, e.g., to mark standards. |
| [↓] | PROGRAMMING SERIES: 01<br>2 LABLED VIAL 00 | Key in number of the second vial to be labled. A maximum of 8 vials can be labeled this way. |
| etc. | | |
| [ESC] | READY (X.X) STD | Instrument is in Ready status. |

**Table 3-5 SERIES Programming**

Programming the instrument is accomplished via the keyboard. Keys are provided for stepping through program parameters and numeric keys for entering data (refer to paragraphs 3.3.1 through 3.3.3 for description of keyboard). Values are entered into memory when stepping to the next or previous program parameters. Programming parameters and data entries are shown on the Display.

Any valid keyboard action is confirmed by a short sound burst when pressing the key. Erroneous keyboard action is indicated by an intermittent sound burst.

The PRIOR (Priority) programming mode can be selected when instrument is in READY mode as indicated on the Display or in RUN mode when the RUN LED lights.

If instrument is in METH or SERIES programming mode as shown on the Display, press ESC key to call up the READY mode.

To create a PRIOR program, refer to Table 3-6.

## 3.4 F (Function) KEY

Five special functions can be selected using the F Key in combination with a code. The special functions are as follows:

- **Remote Control**
- **Erase Method**
- **Proceed**
- **System Gold Network**
- **CCT Column Oven On-Off**

These special functions are discussed in the following paragraphs.

### 3.4.1 Remote Control

Remote Control can be initiated when instrument is in READY mode by pressing **F** followed by **3**. Display will show REMOTE CONTROL. When instrument is in Remote Control mode, it can be controlled by an external controller using Next Vial Input, Next Inj. Input, Stop I/O. Refer to CONTROL I/O pin-outs connections (paragraph 2.4.1( for details).

READY mode is restored by pressing **F** followed by **3.**



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**Symbols:** [⇓]**Next Step,** [⇑]**Previous Step,** [↓]**Substep**

| [ ] KEY | DISPLAY | OPERATOR |
|---|---|---|
| | READY (X.X) STD. | No action required;<br>or<br>Actual rundata (usually be the case when a priority sample must be programmed). |
| [PRIOR ] | PROGRAMMING PRIORITY<br>METHOD NUMBER 001 | Default value. Key in number of Method to be used for processing of the priority sample. |
| [⇓] | PROGRAMMING PRIORITY<br>VIAL NUMBER 01 | Default value. Key in position number of tray where priority sample has been placed. |
| [ESC] | READY (X.X) STD. | If priority was programmed during Run, the priority sample will be processed directly after the current sample being processed.<br>If priority was programmed with instrument in Ready status, the priority sample will be process directly after pressing START key.<br>If for some reason processing of the priority sample should be changed (or not take place at all) after ESC key has been pressed: |
| [PRIOR ] | PRESS C TO CLEAR<br>OR PRIOR TO REPROGRAM | Press [Clear] to erase the priority interruption (no priority will be executed) or press [PRIOR] to change the Priority program. |
| [ESC] | READY (X.X) STD. | As described above for [ESC]. |

**Table 3-6 PRIOR Programming**

### 3.4.2 Erasing Methods

Erasing Methods can be used during METH programming only. Pressing **F** followed by **CL** will permit you to erase the entire Method. Display will show:

METHOD.....XXX
CL = ERASE.....

Press **CL** key to erase Method XXX completely. All parameters will be set to default values. Press any other key when Method XXX is not to be erased.

### 3.4.3 Proceed

The Proceed function will allow you to accelerate the sequence of events as programmed for a certain analysis. It can be used during RUN only and is actuated by pressing **F** followed by **1**. The sequence of events for a regular injection is:

1. Search vial
2. Flushing (the sample line)
3. Loading (filling the loop)
4. Analysis time

Pressing **F** followed by **1** during one of these events as shown on the Display will immediately start the next event, i.e., the analysis time of the sample being processed is stopped and the next injection from the same vial or from the next vial will start immediately.

During programming, several messages may appear on the Display. These messages are meant to inform you of unusual or faulty parameter values or system errors. Refer to paragraph 3.5.2 for description of messages.

### 3.4.4 System Gold Network

To switch from keyboard control to System Gold Network control, press **F** followed by **4**. The Autosampler must be connected to the System network to operate in this mode. The Autosampler can be taken off of the System Gold network by turning Autosample off then back on. Refer to the System Gold Operations Manual for instructions on operating in this mode.

### 3.4.5 CCT Column Oven On-Off

To turn the column oven on or off and to directly set it to a temperature, press F followed by 8. The CCT option must be installed for this function to work. If it is not installed, you will hear two short sound bursts.


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Press the [⇓] to select CCT ON (1) or CCT OFF (2). If the CCT is off, no temperatures in the methods will be used.

Press [⇓] to set an initial temperature. The oven will begin heating to that temperature. Note that temperatures set in the methods will override this setting. Press ESC to finish programming the CCT on-off.

## 3.5 AUTOMATIC SAMPLE PROCESSING

Proper operation of the instrument can be achieved through proper installation and programming techniques. If there is some question about installation, review your installation procedures as discussed in Section 2 of this manual.

The programming techniques necessary to operate the instrument are discussed in paragraph 3.3. Note that during operation, automatic processing can be interrupted and/or redirected at any time. Refer to paragraph 3.5.1.

When operating the instrument, messages may appear on the Display to make the operator aware of actual, special, or unwanted conditions of the instrument or its program. Refer to paragraph 3.5.2 for listing of display messages.

Proceed as follows to setup and operate the instrument.

1. Switch on Power to instrument. Power switch is located on lower right area of rear panel. Display should indicate READY (X.X). The "X.X" represents the software version installed in your instrument.

**NOTE**

**If Display shows POSITIONING NEEDLE, the pneumatics air supply has not been turned on. Turn on and adjust as outlined in paragraph 2.5.**

2. Load tray with sample vials. Refer to paragraph 2.9.

**NOTE**

**Instrument is checked out using IPA prior to shipment. If sample solvent is not compatible with IPA, place a vial with an intermediate solvent as first vial of the first Series. Alternately, use same vial but select first Series with a Method program including flush volume 99, injection volume (equivalent to full loop), and analysis time 00.00.**

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3. Press METH key to enter Method programming. Program as required for your application. Refer to paragraph 3.3.1.

4. Press SERIES key to enter Series programming. Program as required for your application. Refer to paragraph 3.3.2.

5. Make sure LC equipment is set up and operational, i.e., solvent flow rate established, detector on and stabilized, etc. Note that during READY status, instrument injection valve directs solvent flow from pump through sample loop and out to column.

6. Press START key to start automatic processing. The RUN LED will light. When processing is complete, the instrument automatically returns to READY status. While in READY status, you can prepare the instrument for its next series of runs.

### 3.5.1 Interruption And/Or Redirection Of Automatic Processing

Automatic processing can be interrupted and/or redirected as follows:

**HOLD.** Pressing HOLD key stops the analysis time counter and prevents new injections from occurring. HOLD LED indicates when HOLD is activated. Note that any analysis in progress will continue. HOLD is useful when you want to increase the time before the next injection without reprogramming from the keyboard. Pressing HOLD a second time will again start the analysis time counter where it left off.

**RESET.** Pressing RESET key initiates READY status immediately. RESET LED indicates when RESET is initiated. Series programs are not deleted. If the START key is pressed, Series programs will be processed as previously programmed, starting at the first vial of the first Series.

**PRIOR** (Priority). Pressing PRIOR key interrupts automatic processing so a priority sample can be processed. PRIOR LED indicates when PRIOR is activated. The priority sample is processed after processing of the current sample is completed. Refer to paragraph 3.3.3 and Table 3-6 for details on programming and using the PRIOR capabilities.

**Remote Control.** When placed in Remote Control, such functions as Next Injection and Next Vial can be initiated. It is not necessary to put the Autosampler in Remote Control in order to use the Freeze Input. Refer to paragraphs 3.4.1 and 2.4.1 for details.

**Proceed.** Pressing the **F** key followed by a **1** during analysis time will immediately initiate next injection. Refer to paragraph 3.4.3 for further details.


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### 3.5.2 Display Messages

Display messages will appear on the Display to make the operator aware of actual, special, or unwanted conditions in programming or operating the instrument. These messages are discussed as follows:

***MESSAGES AFTER POWER IS TURNED ON***

MEMORY ERROR.....
ALL METHODS ERASED.....

Memory failure makes methods as stored in memory unreliable; all methods are erased (parameters set to default values). This can happen when the memory back-up battery is weak or dead. This can occur if the instrument is not used for an extended period of time. Strike any key to initiate READY status and reprogram Methods.

STD (0) OR RAC (1).....
.....

Press **0** key to select Standard mode or **1** to select RAC mode. Appendix C describes the RAC option.

Pressing **0** will cause the Display to show:

READY (X.X).STD.....
.....

Pressing **1** will cause the Display to show:

READY (X.X).RAC.....
.....

Note that "X.X" indicates the software version

***MESSAGES DURING RUN***

During automatic processing, the display shows the programmed instruction being executed with relevant Series, Method, vial, and injection numbers.

Example: VIAL NR 01 FLUSHING ...
SERIES 01 METH.001 INJ.

***SPECIAL MESSAGES DURING RUN***

FREEZE.....
.....

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CCT NOT READY.....
.....

Injection will not proceed until the oven temperature reaches the set point.

FREEZE input of instrument has been activated (refer to paragraph 3.4.1).

VIAL 97 NOT PRESENT.....
.....

Satellite vial is not present or not positioned properly. Place/position satellite vial; message will disappear.

***SPECIAL MESSAGES DURING***

FLUSHED LOOP.....
.....

Volume entered for injection volume equals loop volume. Instrument will take 1.5 x loop volume out of the sample vial to fill the loop quantitatively.

LOOP VOLUME TOO SMALL.....
.....

Volume entered for injection volume is greater than 60% of loop volume (and not equal to loop volume). Quantitative injection of this volume is not possible with this loop. Enter smaller injection volume or replace sample loop at injection valve and enter relevant value for LOOP VOLUME in the actual method.

***SYSTEM ERROR MESSAGES***

HORIZONTAL ARM.....
MOVEMENT FAILURE.....

Needle arm is obstructed or micro switch defective. Call service engineer.

VERTICAL ARM MOVEMENT...
FAILURE.....

Needle arm does not reach upper position. Check air pressure connection (60 to 80 psi) and check for obstruction. If both ok, call service engineer.

TRAY ROTATION.....
FAILURE.....



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Sample tray obstructed. Check air pressure connection (60 to 80 psi) and check for obstruction. If nothing can be found: call service engineer.

DIGITAL DISPENSER.....
SYSTEM FAILURE.....

Digital dispenser not functioning. Check tightness of the counterblock adjustment. If ok, call service engineer.

### 3.5.3 Priority Interruption Of Run

If automatic processing must be interrupted to process an emergency (priority) sample, the RUN can be interrupted to allow for processing of the priority sample. The position and Method used is programmable. Proceed as follows:

1. Place priority sample vial in any tray position or at satellite position (position no. 97).

**NOTE**

**If the priority sample is to be processed according to a Method that does not exist in the program file, program a Method before proceeding.**

2. Press PRIOR key and select program to be executed.

3. Press ESC key to enter PRIOR mode. PRIOR LED will light indicating entry of Priority mode of operation.

Processing of the priority sample occurs immediately after completion of analysis of the sample being processed at the moment of entering PRIOR mode.

When processing of the priority sample starts, the Display will flash INITIATE PRIORITY, followed by actual data corresponding to the execution of the PRIOR program (showing PRIOR instead of "X" SERIES on the lower Display line).

After analysis of the priority sample is complete, the instrument automatically returns to the RUN program, resuming from the point of interruption. If the system needs equilibration time before the RUN program continues, press HOLD key during analysis time of priority sample to allow for change and equilibration.



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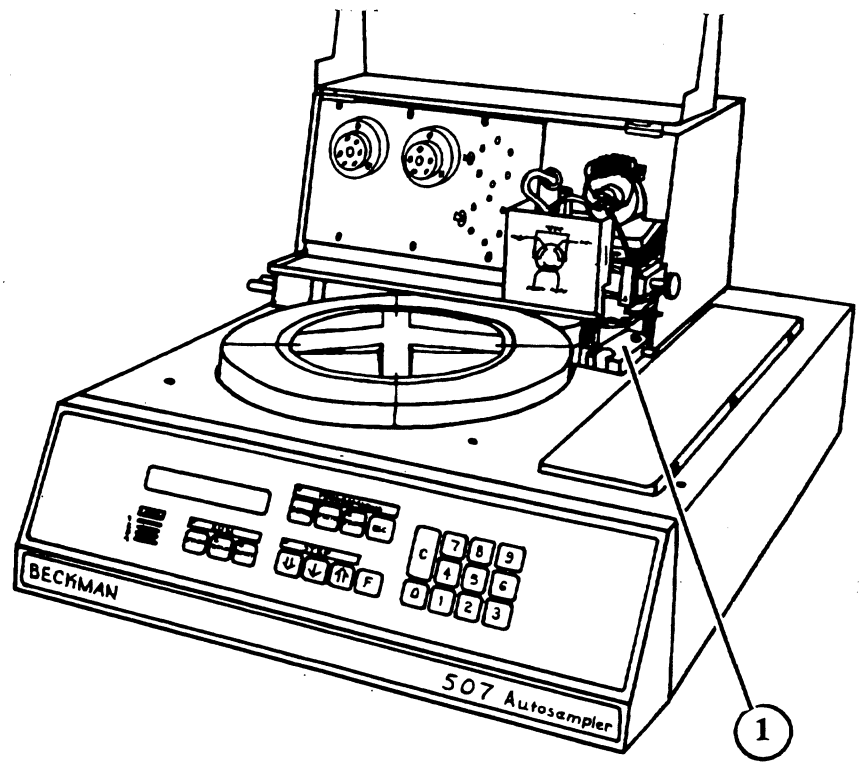
### 3.5.4 Using The Satellite Position

Apart from the 96 sample vial positions in the sample tray, there is one extra "off tray" position called the satellite position. Refer to Figure 3-3 for location of satellite position.
A special large volume vial can be placed in this position. Pull satellite lever counter-clockwise. Place a capped satellite vial and push lever back into its original position.

The satellite position is incorporated in sample processing when the INTERVAL value of the METH program being executed is greater than zero.

*Example*

INTERVAL value = 5: This means that after every 5 sample vials, the needle arm turns to the satellite position.




1. Satellite Vial Position with Holder

**Figure 3-3 LocationOf Satellite Vial Position**


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You can only program the satellite position using METH 99. METH 99 is referred to as the Satellite Method.

The Satellite position can be used for various purposes. Two examples would be needle wash or calibration standard. These techniques are described in paragraphs 3.5.5 and 3.5.6 respectively.

**NOTE**

**You cannot use needle wash if satellite vial contents are used as a calibration standard solution.**

### 3.5.5 Using Needle Wash

You can incorporate needle wash into your Method Program via the INTERVAL parameter. Whenever the INTERVAL parameter is executed, METH 99 is called and the needle wash is executed. Needle wash can be called after each sample vial or a series of sample vials.

The following example illustrates how you would set up the programming to wash the needle after *each* sample vial.

1. Place wash vial in Satellite position. Refer to Figure 2-5 for location of Satellite position.

2. Program Satellite Method as follows:

   METHOD NO.....99
   ANALYSIS TIME...00
   EVENT METHOD...Enter relevant value if desired.
   LOOP VOLUME.....Enter relevant value.
   INJ.VOL.....000
   FLUSH VOLUME ...Enter relevant value (30 - 99 µL).

**NOTE**

**It is important that "000" be entered for INJ. VOL when performing a needle wash. If not, an injection of specified volume will be made.**

3. Program your Analysis Method for sample injection followed by needle wash as follows:

   METHOD NO. ....."X" (1 - 98 or 100 - 109)
   ANALYSIS TIME .....Enter relevant value.



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LOOP VOLUME.....Enter relevant value.
INJ. VOLUMES.....Enter relevant value (1 - 9).
FLUSH VOLUME .....Enter relevant value.
INTERVAL VALUE ....1 (specifies wash after every vial)

**NOTE**

**When programming a SERIES (one or more samples) incorporating needle wash, make sure you enter METH "X" in the SERIES program, not METH 99.**

### 3.5.6 Using Calibration Standard

You can incorporate a calibration standard into your Method Program via the INTERVAL parameter. Whenever the INTERVAL parameter is executed, a routine called Satellite Method (METH 99) is called and the external standard is executed. The calibration standard can be called after each sample vial or a series of sample vials as specified in the Interval Value in the Method..

The following example illustrates how you would set up the programming to inject a calibration standard after every second sample vial.

1. Place wash vial in Satellite position. Refer to Figure 2-5 for location of satellite position.

2. Program Satellite Method as follows:

   METHOD NO.....99
   ANALYSIS TIME...00
   EVENT METHOD...Enter relevant value if desired.
   LOOP VOLUME.....Enter relevant value.
   INJ.VOL.....Must enter an injection volume.
   FLUSH VOLUME ...Enter relevant value (30 - 99 µL).


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3. Program your Analysis Method for sample injection (two in this example) followed by external standard injection.

   METHOD NO. ....."X" (1 - 98).*
   ANALYSIS TIME .....Enter relevant value.
   EVENT METHOD.....Enter relevant value if desired.
   LOOP VOLUME.....Enter relevant value.
   INJ. VOLUMES.....Enter relevant value (1 - 9).
   FLUSH VOLUME .....Enter relevant value.
   INTERVAL VALUE ....2 (1 - 96 available)

**NOTE**

**When programming a SERIES (one or more samples) incorporating an external sample, make sure you enter METH "X" in the SERIES program , not METH 99.**

### 3.5.7 Extended Shutdown

If the instrument is not to be used for an extended period of time, flush needle and injection valve with a solvent appropriate to the sample/mobile phase last used. Also, loosen the counterblock adjustment whenever the autosampler won't be used for several days (24 hrs). If you loosen the counterblock adjustment, it will be necessary to operate the DDS pump for 800 cycles to return the flexibility of the tubing in the pump. This can be done by executing several dummy injections.

**⚠ CAUTION**

**Never allow a buffer or salt solution to remain in the system during extended shutdown because crystalization can occur. If salt buffers have been in use, then flush with water, not IPA which may cause precipitation. Also, it is preferable that the counter block of the DDS is loosened by two turns counter-clockwise. Refer to Figure 4-1 for location of counter block.**

* The µL Pickup Method (METH 100 - 109) cannot be used since satellite position does not contain mobile phase.



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# Section 4
# Principles Of Operation

## 4.1 GENERAL DESCRIPTION

This section of the manual will point out the various components or systems that are part of the 507 Autosampler. Some components are self-explanatory. Other components or systems, e.g., injection system and digital-dispenser system, require further explanation. The following paragraph will cover principles of operation of these systems.

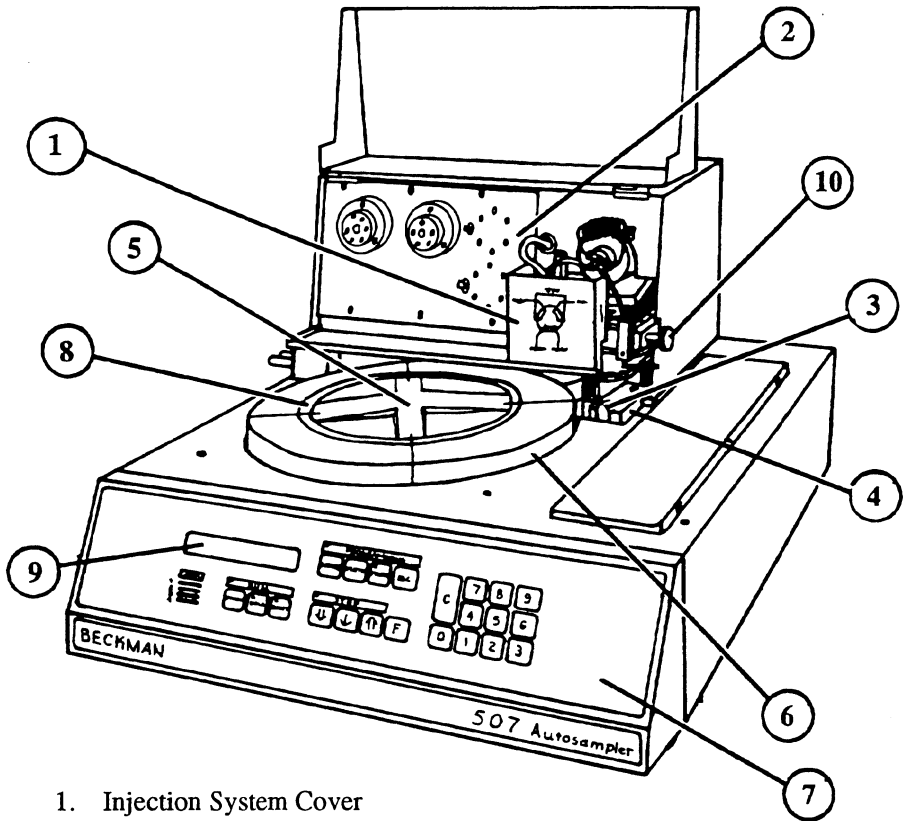
Refer to Figure 4-1 for general view of instrument and the location of components and systems.

## 4.2 COMPONENTS AND SYSTEMS

Central to the operation of the 507 Autosampler is the unique valve switching, and digital-dispenser system (DDS), and injection system. This system makes possible the injection of sample volumes in a simple and reproducible manner.

The injection system comprises an injection valve with sample loop and the digital-dispenser system pump and sample needle. Refer to Figure 4-2 for location of major components comprising the injection system. This system permits 3 different injection Modes: Flushed Loop, Partial Loop, and µL Pickup. The injection and digital-dispenser pump systems will be discussed in the following paragraphs followed by a discussion on the injection Methods.


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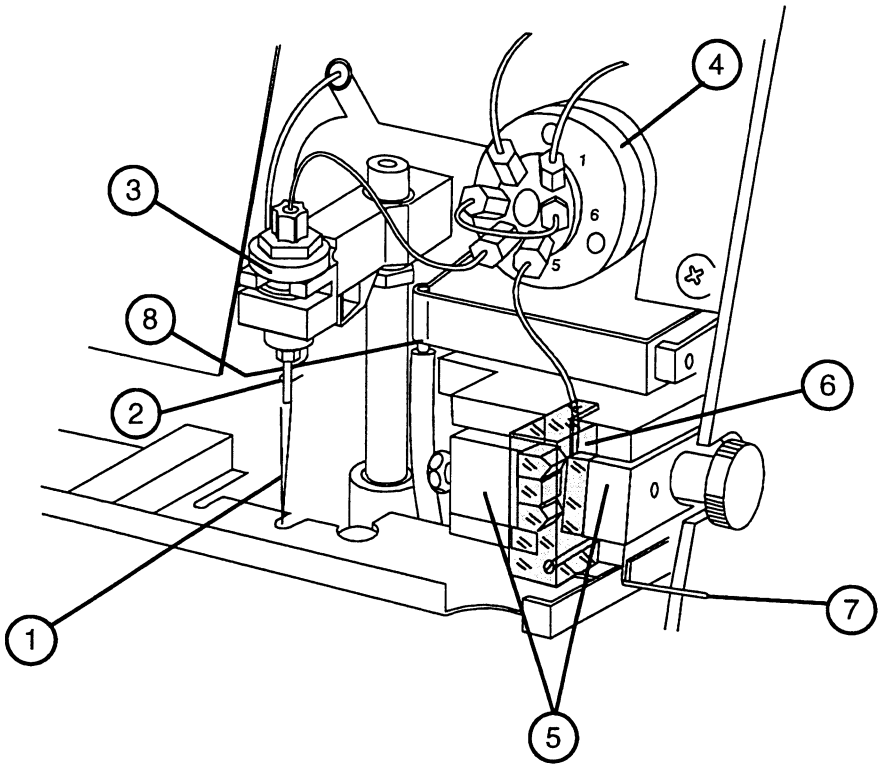



1. Injection System Cover
2. Option Space (ssv)
3. Satellite Position
4. Satellite Position Lever
5. Grip-Manual Sample Transport
6. Sample Tray (4 segments)
7. Keyboard
8. Sample Position Indicator
9. Display
10. Dispenser Counter Block Locking Nut

**Figure 4-1 507 Autosampler**



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1. Sample Needle
2. Air Needle
3. Needle Adjustment Screw
4. Injection Valve
5. Digital Dispenser System
6. Pump Tubing Cassette
7. Dispenser Drain
8. Leakage Drain for Injection Valve

**Figure 4-2 Components of Injection System**

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### 4.2.1 Injection System

The injection system comprises a 6-port Rheodyne 7010 injection valve, a digital-dispenser (finger pump) system, and an injection needle. The air actuated injection valve has two positions: Inject and Load. In Load, the sample is pumped from the vial through the sample needle into the sample loop via the digital-dispenser pump. When the valve switches to the Inject position, the sample is flushed from the sample loop and into the column via the solvent flow from the pump.

The motor-driven digital-dispenser pump uses three fingers to achieve pumping action. As shown in Figure 4-3, Fingers 1 and 3 act as inlet and outlet pinch valves respectively, and Finger 2 provides the fluid displacement valve.

The sample is pumped through a tube made of a fluor-elastomer (Dupont Kalrez®). The Kalrez® tube is subject to abrasion so it is surrounded by a silicon rubber sleeve, which is in contact with the pump fingers. The result is a long-lived, reliable system.

This pump is key to proper operation of the injection system. Note that the stroke of finger number 2 can be adjusted to change displacement value of the pump.

The pump is factory calibrated to 1 µL per stroke. The volume dispensed is always a multiple of one stroke volume.

**NOTE**

**The Kalrez® tubing will develop a "memory" if the pump is not run for several days. This will cause the DDS pump to fail to deliver reproducible injections, especially in partial loop or µL pickup modes. The tubing will return to normal after running the pump for about 800 strokes. To avoid this situation, loosen the counterblock adjustment nut when the autosampler is not going to be operated for more than 12 hours.**



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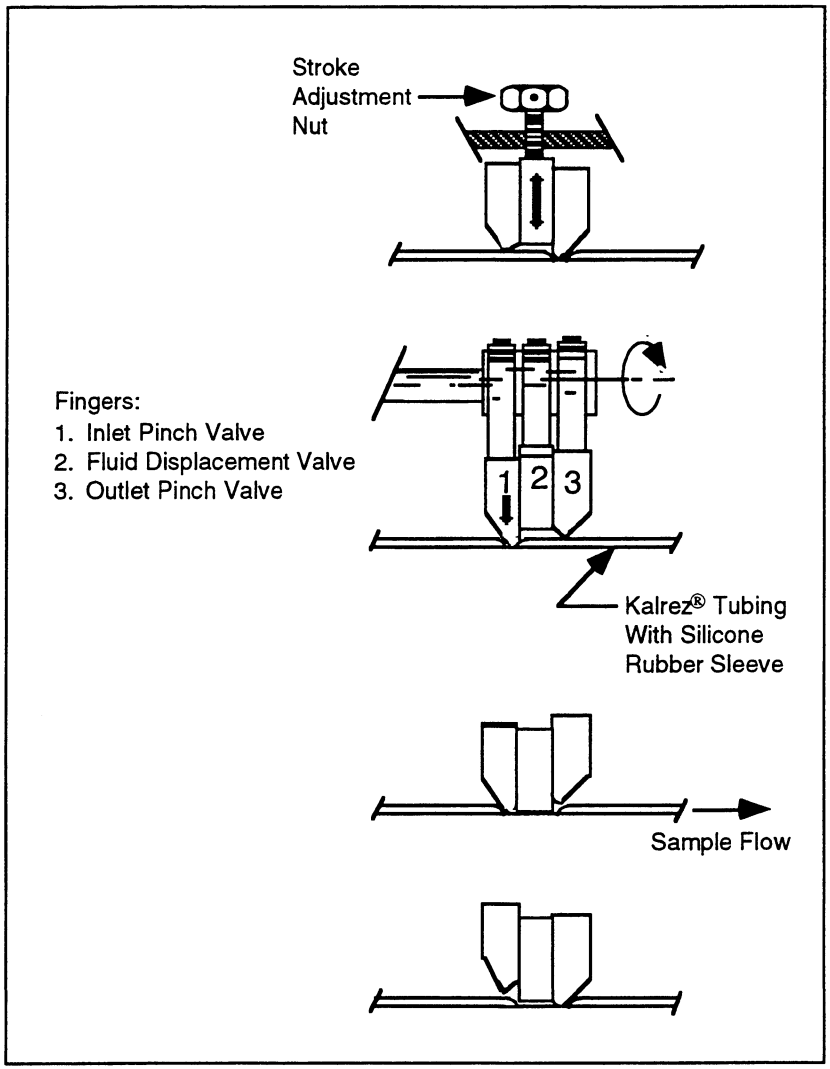



**Figure 4-3 Digital-Dispenser Pump Stroke Sequence**



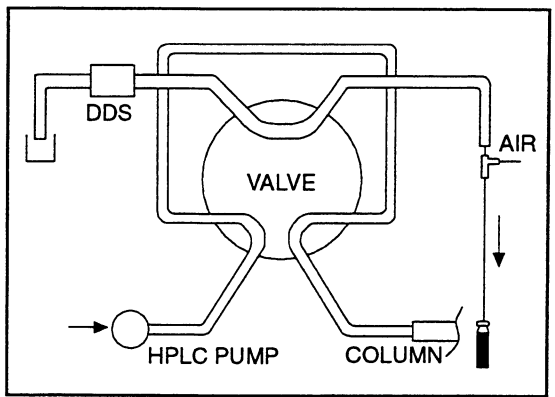
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### 4.2.2 Injection - Flushed Loop/Partial Loop

In Flushed Loop injection, the sample loop is completely (quantitatively) filled with sample resulting in extremely good reproducibility (better than 0.5 %). An illustrated description of this Method is shown along with text in Figures 4-4A through 4-4F.

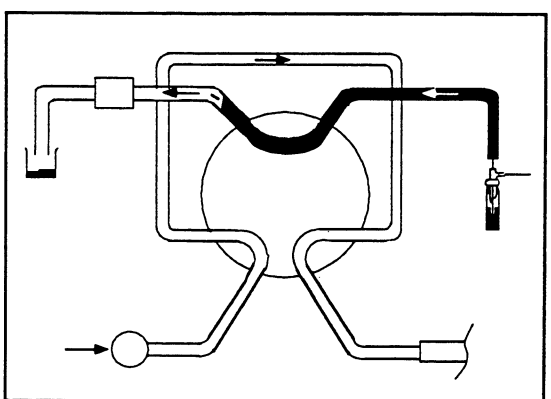
In Partial Loop injection, the sample loop is partially filled with sample, permitting low sample loss and programmable injection volume. This Method is also illustrated in Figures 4-4A through 4-4F.

**Figures 4-4A through 4-4F Flushed Loop/Partial Loop Method**




4-4A

Initially, the Injection valve is in Inject position. First, a 3 µL air gap is created to form a boundary between the liquid in the needle and the sample. Next, the co-axial needle pair is inserted into the sample. Air pressure, applied on the sample through the outer air needle, ensures that a vacuum is not formed during sample withdrawal.

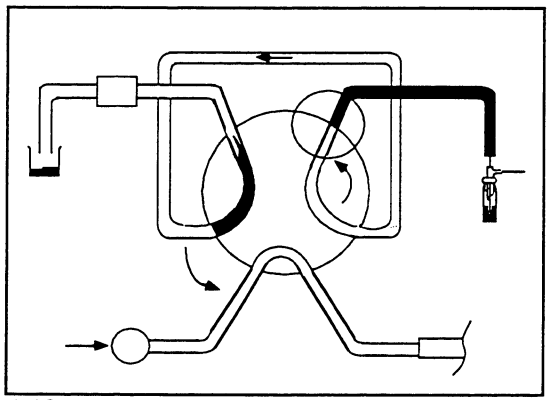


4-4B

Dispenser pump withdraws the specified "flush volume" from sample vial to fill sample line with sample and to remove previous sample.

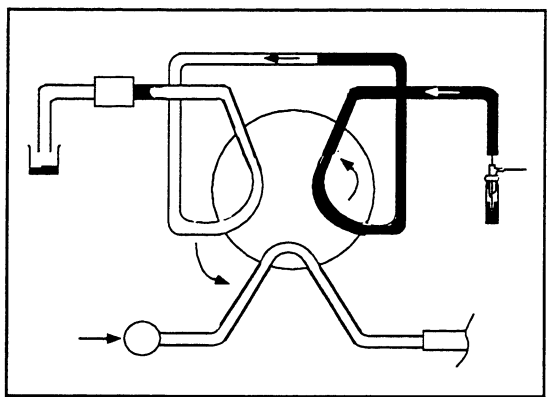


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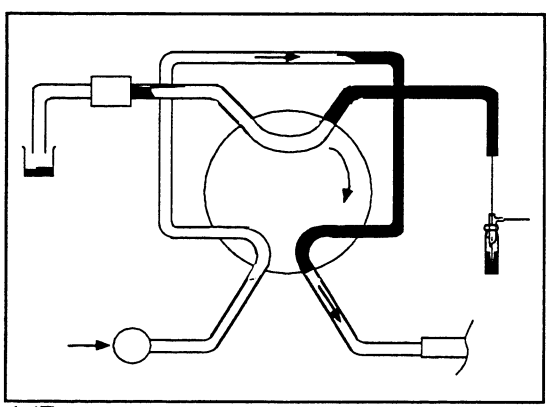
4-4C

Injection valve switches to Load position, placing a "sharp" sample front at the very inlet of the sample loop.



4-4D

*Partial loop fill*: dispenser pump transports selected injection volume into the sample loop. *Flushed loop*: dispenser pump transports 1.5 x loop volume through the loop to fill the loop quantitatively.

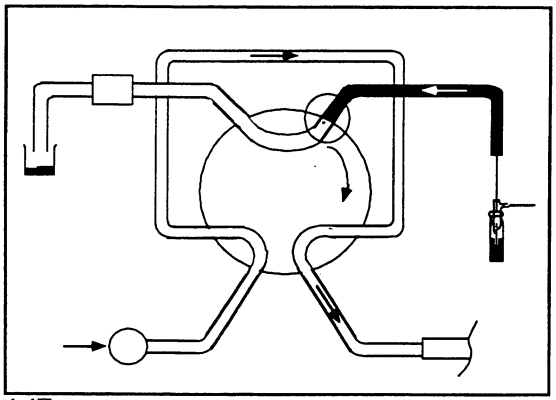


4-4E

Injection valve switches into Inject position. Sample loop is now part of the HPLC mobile phase flow path. Sample is transported to the column. Analysis time starts. If one injection is to be made out of each sample vial, needle pair withdraws after analysis time and sequence **A** to **E** starts over. If more than one injection is to be made out of the same sample vial, see **F**.



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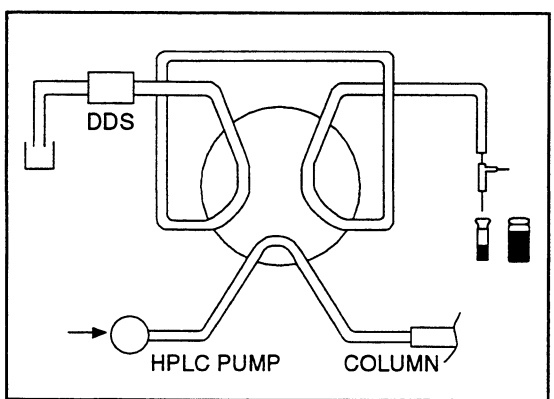
4-4F

After analysis time with subsequent injections from the same vial, the dispenser pump withdraws a pre-flush volume (one-half of the specified flush volume) to compensate for diffusion of mobile phase which occurs in front of the sample line during analysis time. Diffusion is caused by the rotor grooves in the injection valve. Sequence continues at **B**.

### 4.2.3 Injection - µL Pickup

In µL Pickup, the sample volume withdrawn from the vial is transported into the loop with mobile phase from the satellite vial. No sample loss occurs. As little as 1 µL may be injected from 5 - 10 µL sample. An illustrated description of this Method is shown along with text in Figure 4-5A through 4-5E.

**Figures 4-5A through 4-5E µL Pickup Method**

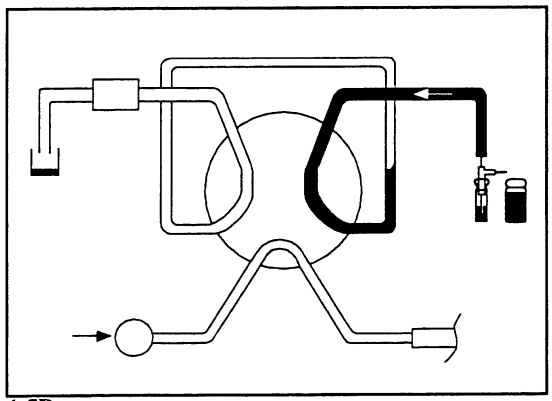



4-5A

Injection valve is in Load position. The coaxial needle pair is inserted into the sample. Air pressure, applied to the sample, ensures that a vacuum is not formed during sample withdrawal.

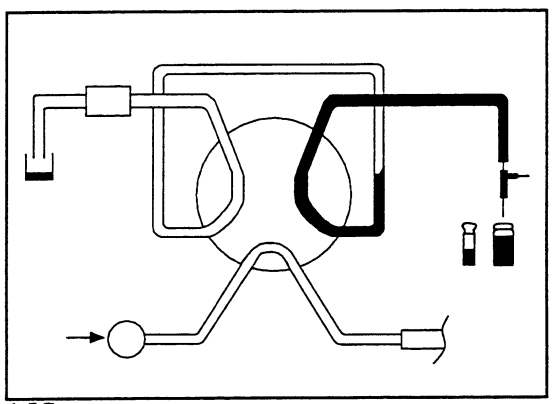


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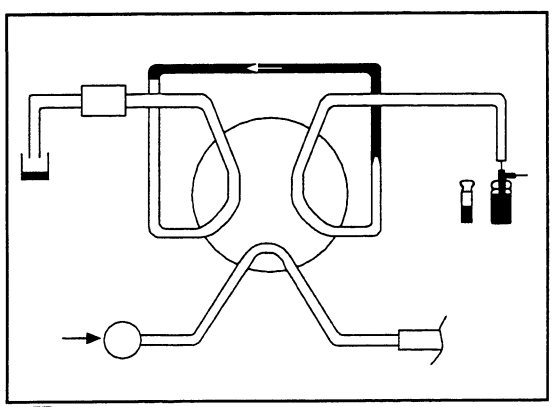
4-5B

Dispenser pump withdraws programmed injection volume.



4-5C

Needle pair is withdrawn. Needle arm now moves to satellite position and needle pair is inserted in satellite vial containing mobile phase.

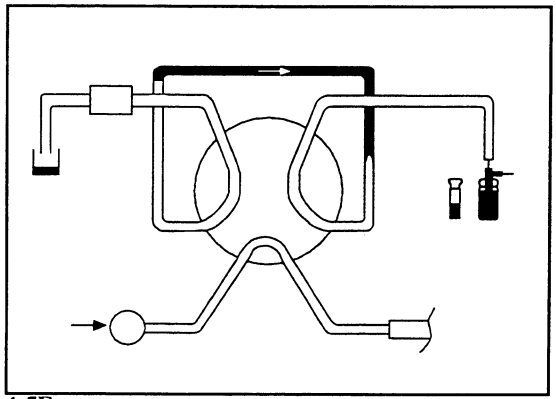


4-5D

Dispenser pump withdraws 20µL mobile phase from the satellite vial to transport the sample quantitatively into the loop.



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4-5E

Injection valve switches into Inject position. Sample is swept onto the column. Next injection(s) are made starting over at **A** and can be either from the same or another vial.



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# Section 5
# Calibration/Adjustments

## 5.1 GENERAL INFORMATION

This section of the manual will outline procedures for calibration and/or adjusting the 507 Autosampler. These procedures can be performed by the customer. The procedures covered are listed as follows:

- Adjusting and Calibrating the Digital-Dispenser Pump System (DDS)
- Adjusting the Needle/Tray Position

## 5.2 ADJUSTING AND CALIBRATING THE DIGITAL-DISPENSER PUMP

The following paragraphs discuss adjusting and calibrating the digital-dispenser pump. If you have the RAC option installed, refer to Appendix C for proper adjustment procedures.You must first remove the injection system cover (See Figure 5-1), then the injection valve drip plate located just below the injection valve (See Figure 5-2) before the following procedures can be carried out. Be sure to reinstall these items before operating the instrument.

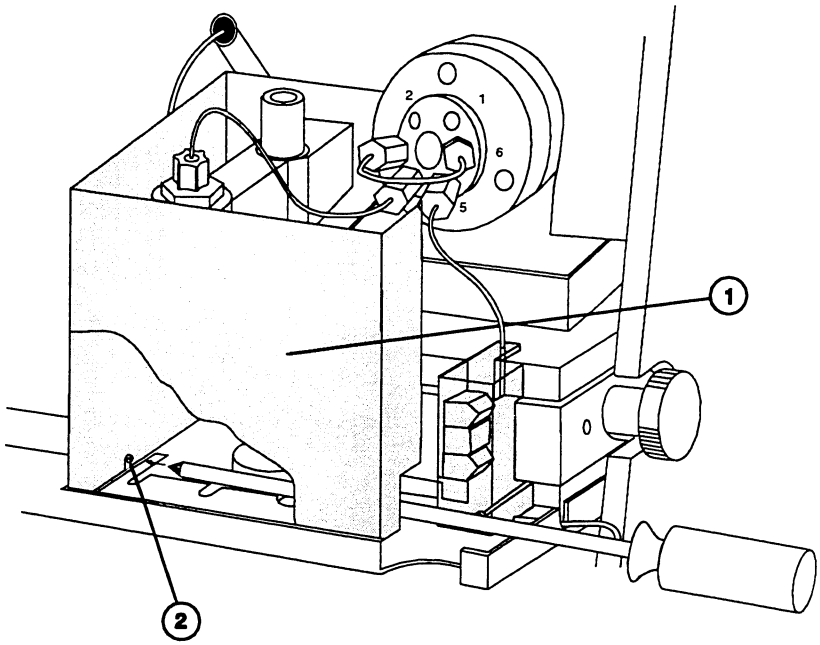
**⚠ WARNING**

**When the needle shield is removed, the needle is exposed and personal injury may occur if an injection occurs while working in this area.**

**NOTE**

**Before performing the following adjustments and calibrating procedures, make sure there is no internal leak in the injection valve. Adjusting the DDS may just compensate for the leak. A leak is indicated when a drop forms on the end of the needle. This is caused by a leaking rotor seal. See Section 7, paragraph 7.3 for rotor seal replacement instructions.**

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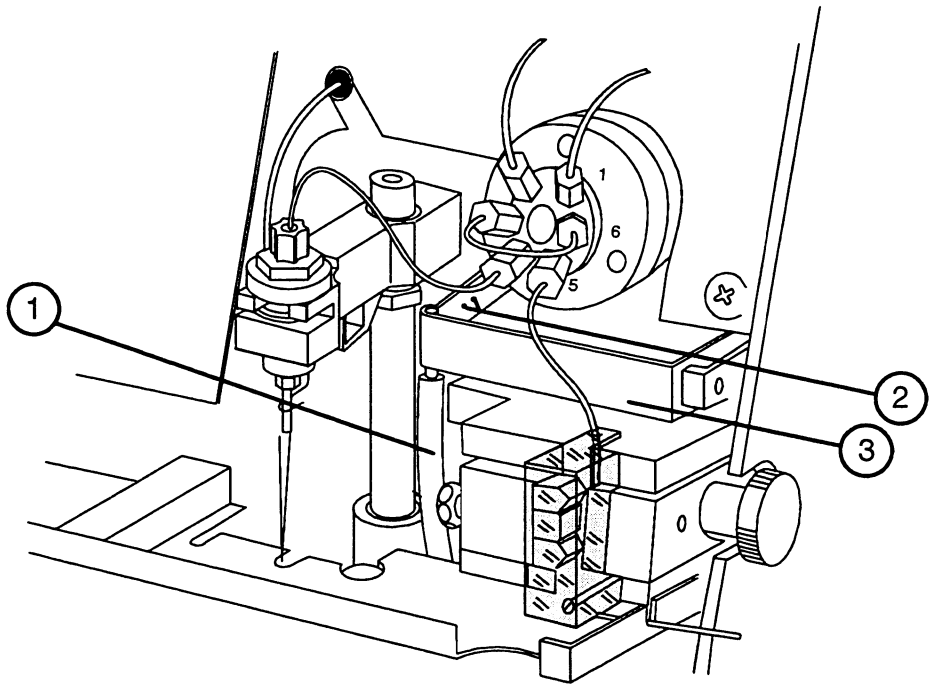



1. Injection System Cover
2. Loosen screw with screwdriver then lift up and remove Injection System Cover

**Figure 5-1 Removing Injection System Cover**



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1. Disconnect Drain Tubing
2. Remove Screws
3. Injection Valve Drip Plate

**Figure 5-2 Removing Injection Valve Drip Plate**



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### 5.2.1 Disc Position

Set the pump rotation disc as shown in Figure 5-3 when the finger pump is in the stop position as shown in view **A** of Figure 5-4. The outlet finger is lifted, closing the tubing. At the same time, the inlet and displacement finger are not lifted but are resting against screws **A** and **B** as shown in Figure 5-5. It will help to split the difference between when the outlet finger begins to close, and 2) when it begins to open, to set this disc position.

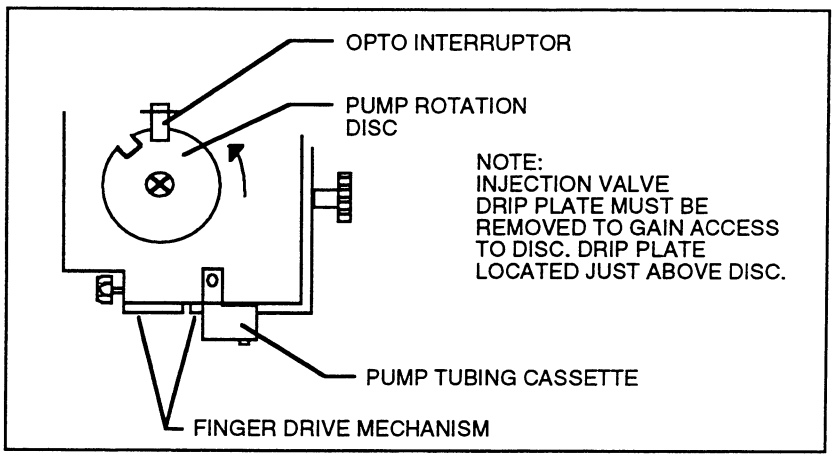



**Figure 5-3 Dispenser Pump Disc Position**

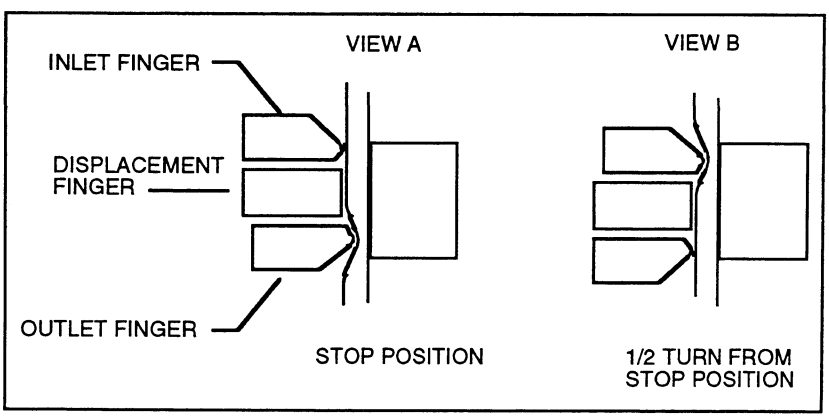



**Figure 5-4 Finger Stop Positions**


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### 5.2.2 Lift Height - Inlet/Outlet Fingers

The lift height of the inlet and outlet fingers should be adjusted to approximately 0.5 mm. This adjustment is not critical and need not be done often. But if the lift height is much large, e.g., 1 mm, then the pump flow is pulsating much more then necessary and can cause poor reproducibility. As shown in Figure 5-5, adjust lift height as follows:

1. Program a 100 µL injection volume, 9 injections per vial, and an analysis time of 0.

2. Start Method.

3. Turn adjustment screws **A** and **C** just until the fingers just stop moving.

4. Back out each screw, i.e., **A** and **C**, with one (360°) counterclockwise turn.

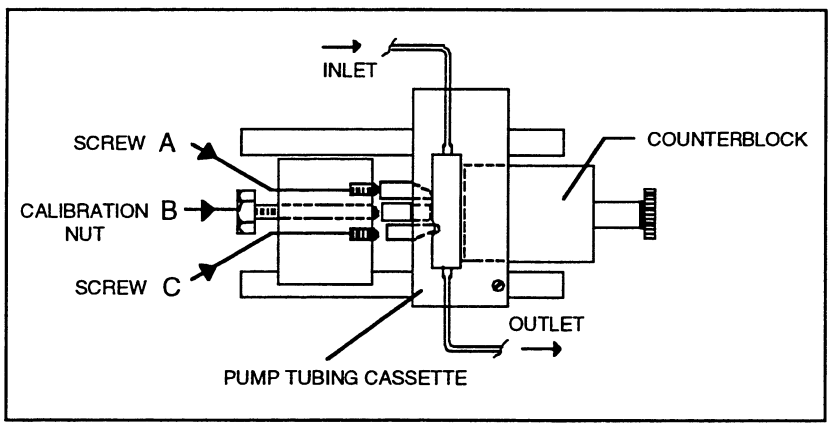



**Figure 5-5 Finger Lift Height Adjustments**



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### 5.2.3 Counterblock Adjustment

Checking the correct closing of the tubing via the outlet finger is possible by placing an empty and newly capped vial in the tray. While the needle is in the sample vial, the vial is pressurized. No air bubbles should be seen when you hold the waste outlet in a vial with water.

If air bubbles are seen, the counterblock adjustment must be made. Alternately, you can manually check the counterblock adjustment by turning the dispenser pump disc a half turn to check that the inlet finger closes the tubing as shown in Figure 5-4 view **B**. Referring to Figure 5-6, adjust counterblock as follows:

1. Place an empty new capped vial in the tray.

2. Immerse waste outlet of the dispenser pump in a cup of water.

3. Loosen set screw **D** and locking nut **E** two turns counterclockwise.

4. Program a 30µL flush, 20µL injection volume, and 90 minute analysis time.

5. Start the Method.

6. When the needle arm is down, the dispenser pump stops pumping (stop position shown in Figure 5-3). Close the counterblock slowly with locking nut **E** until the airflow just stops in the cup of water.

7. Turn locking nut **E** 1/8 to 1/6 of a turn clockwise. Now fix this position by turning set screw **D**. The outlet finger now closes the tubing.

8. Check that the inlet finger closes the tubing by manually turning the counter disc 180° (refer to Figure 5-4 view **B**). If the inlet finger does not close the tubing, loosen the fixation nut **D** and turn the locking nut **E** and additional 1/8 of a turn. Check that this additional turn does not slow the rotating speed of the dispenser too much.

**⚠ CAUTION**

**The lifetime of the pump tubing will be reduced significantly if the counterblock is adjusted such that the tubing is pressed very tight.**



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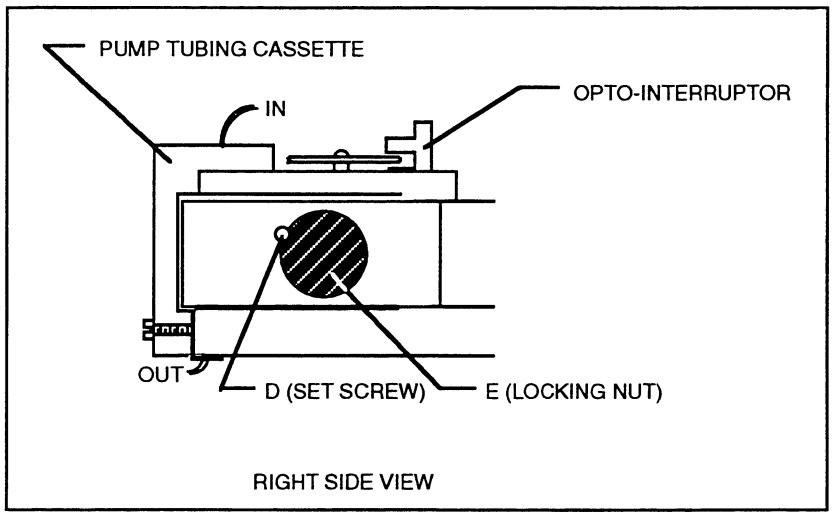



**Figure 5-6 Counterblock Adjustment**

### 5.2.4 Dispenser Calibration

After checking the previously described adjustments and visual inspection of the pump tubing (no mechanical damage of the silicon sleeve which fits into the dispenser), you can check or adjust the stroke volume of the pump (factory adjusted to 1 µL/stroke). Check and/or calibrate pump as follows:

1. Weigh a vial of water. Use the µL Pickup Method to withdraw 20 µL from the vial. Reweigh the vial. Since 1mL of water weighs 1mg, the amount removed can easily be determined, i.e., 20 mg. If the pump is out of calibration, perform step (2), then repeat step (1) until pump delivers 20 µL.

2. Turn calibration nut **B** (refer to Figure 5-5 clockwise to decrease stroke volume and counterclockwise to increase stroke volume. Each full turn equals about 1µL/stroke. Use a small hex-key to turn the screw.

**NOTE**

**Since sample loop volumes can vary by as much as 20%, an accurate partial loop injection may not be proportional to a flushed full loop injection.**

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## 5.3 NEEDLE/TRAY POSITION ADJUSTMENTS

The following paragraphs discuss needle penetration depth and penetration position adjustments.

### 5.3.1 Needle Penetration Depth Adjustment

To keep the dead volume small, needle penetration depth should be adjusted such that the sample needle stops just above the bottom of the sample vial. To make the adjustment, proceed as follows:

1. Turn off main power and disconnect air supply.

2. Place an uncapped vial in position 95 and rotate tray so vial is underneath the needle.

3. Remove tray segment number 1.

4. Move the sample needle down to its lowest position in the vial.

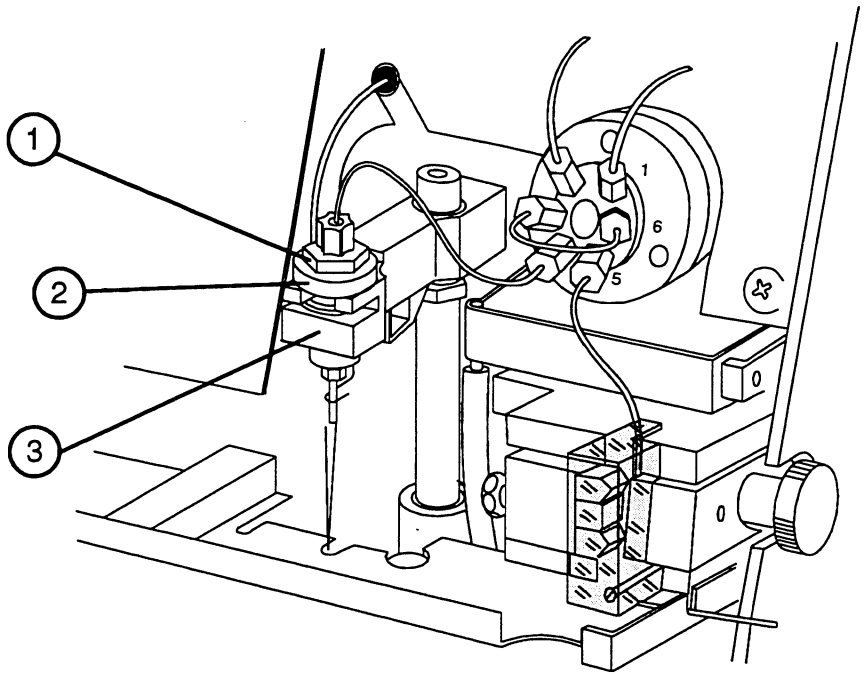
5. Loosen the locking nut. Refer to Figure 5-7 for location of nut.

6. Adjust the penetration depth with the knurled penetration adjustment nut. Clockwise rotation moves the needle up. The needle should stop not less than 1 mm above the bottom of a standard vial and not less than 2 mm above the bottom of a conical vial.

7. Tighten locking nut to secure adjustment.

8. Reconnect air supply.

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1. Locking Nut
2. Penetration Adjustment Nut
3. Needle Arm

**Figure 5-7 Needle Penetration Depth Adjustment**



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### 5.3.2 Needle Penetration Position Adjustment

To adjust the position that the needle penetrates the vial septum, follow the procedure below:

1. Turn on instrument. If not off already, turn off air supply.

2. Insert a few uncapped vials at different positions on the tray.

3. Program a series with one vial (e.g. from 5 to 5).

4. Start the unit. It will stop at the programmed position.

5. Now manually push the needle down and check its position. The needle should be centered in the vial.

6. Adjust the penetration position by moving the entire needle position assembly. Loosen the two screws shown in Figure 5-8. Check needle position at different places (using a one-vial series for the correct stop position). The specification for center accuracy is ± 1mm. It may vary slightly from vial to vial.

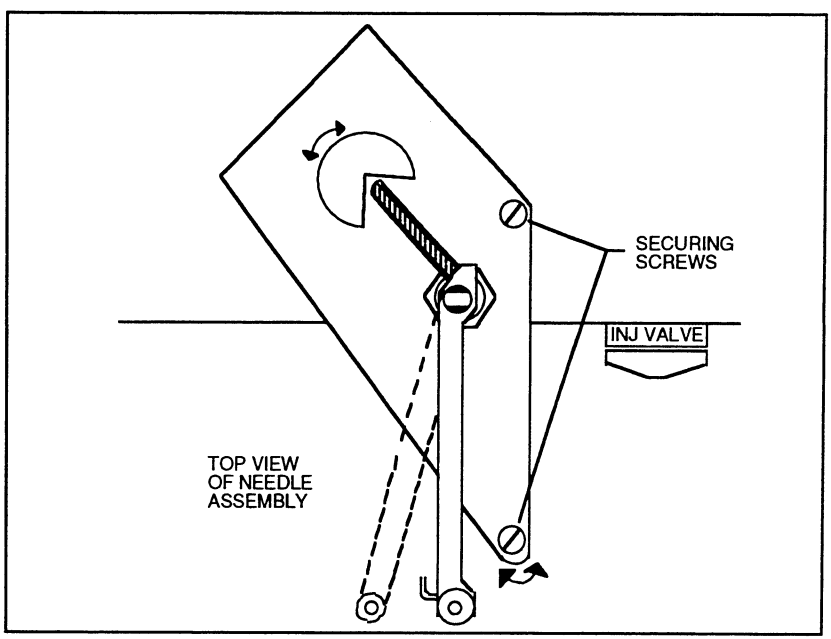



**Figure 5-8 Needle Penetration Position Adjustment**


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# Section 6
# Troubleshooting

## 6.1 GENERAL INFORMATION

This section of the manual covers certain diagnostic and troubleshooting procedures necessary to check instrument performance and diagnose and correct instrument problems. Areas covered in this section include:

- Specification Test
- Tray Positioning
- Error and General Messages
- Troubleshooting Table
- Tracing Blockage/Leakage in Sample Flow Path

## 6.2 SPECIFICATION TEST

It is important to use the following procedure to check the performance of the 507 Autosampler with respect to reproducibility and carry-over. Tests which use chromatographic peaks may not be valid because many other variables are present which can cause apparent injection problems. Referring to Figure 6-1 for test configuration, perform a specification test as follows:

1. Make up a sample of 100 mg/L Uracil in HPLC grade water. The sample should be made fresh no longer than one day before using.

2. Plumb the HPLC system as shown below:

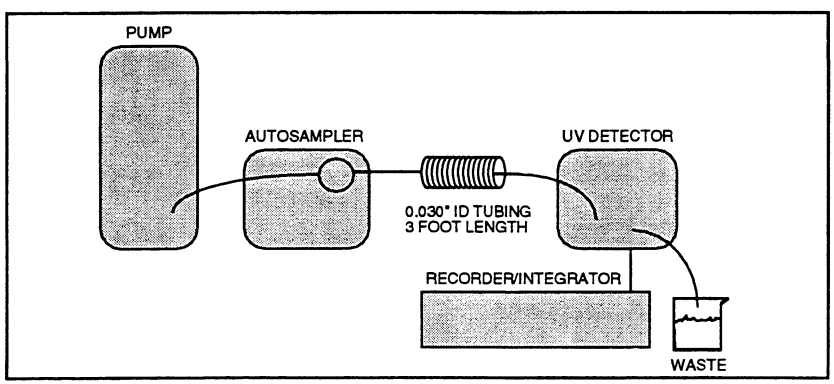



**Figure 6-1 Test Configuration for 507 Autosampler**


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3. Set pump flow to 1.5 mL/min of 100% water.

4. Set detector to 254 nm and about 0.5 AUFS.

5. Set autosampler to 30 µL flush volume, 10 µL injection volume, 3 injections per vial, and a 1 minute analysis time.

6. Fill 5 vials with the Uracil solution and 2 vials with HPLC grade water. Be sure to use new nonpunctured septa.

7. Place the vials in the sample tray in the following sequence i.e., 1 sample vial in position number 1, 2 water vials in positions 2 and 3, and 4 sample vials in positions 4 through 7.

**NOTE**

**If the autosampler has not been used for a long time or has been used with other eluents, then start first with an extra vial filled with water and flush the system thoroughly. Program a 100 µL flush, 8 injections of 100 µL, and 0 analysis time.**

8. Start the system and make the injections. If the test is stopped and restarted for any reason, then recap any vials which were punctured.

Check reproducibility by calculating the coefficient of variation of the 15 peak areas from the first vial and last 4 vials. It should be less than 1% to be within specification.

To calculate the amount of sample carry-over, compare the difference in peak areas between vials 2 and 3 to the average area from vial 1.

The difference between vials 2 and 3 is calculated to eliminate refractive index effects that occur when the injection valve is actuated. Since the sample is not retained in this test, the refractive index peak interferes with the Uracil peak. The peak from vial 2 includes any carry-over plus the refractive index peak. Vial 3 should only include the refractive index peak.

The area of the sample carry-over peak should be less than 1% of the area of vial 1. If the reproducibility or carry-over is not within specification, then:

1. Check that the vials are capped so no air leaks exist.

2. Check for leaks or restrictions in the tubing and connections between the needle and injection valve and injection valve and dispenser pump (see Section 7, paragraph 7.2.1).


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3. Check for a restriction in the sample needle and replace if necessary (see Section 7, paragraph 7.2.1)

4. Replace the injection valve rotor seal (see Section 7, paragraph 7.3.1).

5. Check the dispenser pump adjustment and calibration (see Section 5, paragraph 5.2)

6. Replace the dispenser pump tubing cassette (see Section 7, paragraph 7.2.3).

## 6.3 CHECKING TRAY POSITIONING

Program a series of 1-96 vials. Start with an empty tray. After finding position one, the unit will now search for a vial (reading the tray number) for 1 up to 96.

If for some reason a particular number is not found, the unit will continue searching for that number (indicated by faster stepping) and the missing number will be displayed. The main causes of this problem are:

- The Tray Position PCB is incorrectly positioned.
- One or more of the opto-sensors is failing.

If the unit makes a complete turn and ends with the message READY shown in the Display, every position was found correctly.

## 6.4 ERROR AND GENERAL MESSAGES

Messages may appear on the Display to make the operator aware of actual, special, or unwanted conditions of the instrument or its program. These messages with discussion are listed as follows:

**DIGITAL DISPENSER SYSTEM FAILURE**. Dispenser does not rotate or rotates too slow (or opto-interrupter failure). Reset the machine and adjust the dispenser pump (see Section 5, paragraph 5.2).

**FLUSHED LOOP**. Indicates that, during method programming, the volume entered for injection volume is equal to the loop volume. The autosampler will take 1.5 x the loop volume from the sample vial to overfill the loop.

**FREEZE.** Indicates that the freeze remote input is active. The autosampler will not proceed until the input is deactivated. Controller I/O is held to ground.



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**HORIZONTAL ARM MOVEMENT FAILURE.** After a left/right command the needle arm is not into its correct position within 2 seconds. Release by pressing RESET. Possible causes for this problem: motor, motor relay, or needle-arm not in upper position. Call Beckman Service if pressing RESET does not solve problem.

**LOOP VOLUME TOO SMALL.** An attempt was made to program an injection volume which is too large compared to the known loop volume. After 2 seconds, this message disappears.

**MEMORY ERROR, ALL METHODS ERASED.** Usually follows the Memory error initiating Methods message. Indicates that memory has been erased and all the Methods will have to be reprogrammed.

**MEMORY ERROR INITIATING METHODS.** Appears for 1 second on the Display after switching the power on when the memory check was negative (e.g. battery back-up failure). This message is followed by MEMORY ERROR, ALL METHODS ERASED. Memory is then erased and all methods are initialized with their default values. Release by any key.

**TRAY ROTATION FAILURE.** After a step command the tray does not come into the next stop position within 3 seconds. Release by pressing RESET. Possible causes for this problem: motor, motor belt, motor relay, or needle-arm not in upper position. Call Beckman Service if pressing RESET does not solve problem.

**VERTICAL ARM MOVEMENT FAILURE.** After an up command the needle does not move into to the upper position within 5 seconds. Release by pressing RESET. Possible causes for this problem: air pressure, solenoids, "up"-microswitch. Call Beckman Service if pressing RESET does not solve problem.

**VIAL 97 NOT PRESENT** - Vial 97, the satellite vial, is not present or positioned properly. Clear this message by properly positioning the satellite vial.

## 6.5 TRACING BLOCKAGE AND/OR LEAKAGE IN THE SAMPLE FLOW PATH

The following information provides a method for tracing blockage and/or leakage in the sample flow path.



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As shown in Figure 6-2, the sample flow path comprises:

1. Sample needle
2. Needle connection tubing
3. Injection valve internal passages
4. Sample loop
5. PTFE tubing connecting the injection valve-to-dispenser pump tubing
6. Dispenser pump internal tubing
7. Dispenser pump waste outlet

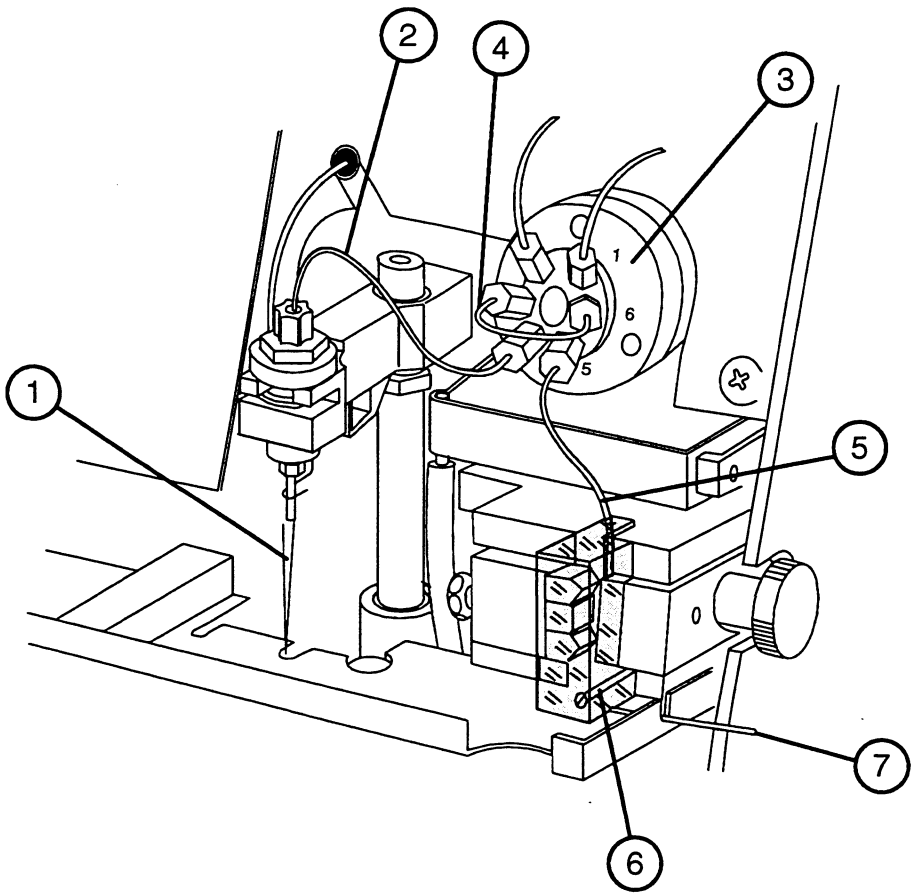



**Figure 6-2 Sample Flow Path**



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If a blockage is suspected in the sample flow path, then make the following checks:

1. Place a vial with pure solvent (water or methanol) in the tray.

2. Loosen the counterblock of the dispenser pump by turning the knurled nut counterclockwise 3 or more times .

3. Program an injection out of the vial.

4. Start the injection. As soon as the needle pair penetrates the septum of the vial, solvent should pour out freely at the waste outlet of the dispenser pump (due to the vial pressure and the lack of restriction from the dispenser pump).

If solvent does not come out of the waste line, a blockage is present. Proceed as follows:

1. Disconnect the silicon air supply tubing at the needle arm by sliding it off the steel capillary.

2. Submerge the loose end of the silicon tubing in water.

3. Start the injection. A vigorous bubbling should occur if the air supply is correct. If not, then the blockage is likely in the air supply to the air needle.

If the air needle supply is ok, then proceed as follows:

1. Reconnect the needle air supply tube to the steel capillary.

2. Place a completely filled *uncapped* vial in the tray and program an injection.

3. Start an injection. A vigorous bubbling should occur. If only a gentle bubbling occurs, then the air needle may be blocked. If the air needle looks okay, then the air passage in the needle arm may be blocked.

If the vial is receiving the proper air supply, then proceed as follows.

1. Disconnect the needle connection tubing at the injection valve port 4.

2. Place a *capped* vial with pure solvent in the tray and program an injection.



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3. Start the injection. When the needle pair goes into the vial, solvent should flow out the loose end of the PTFE tubing. If not, then the sample needle or tubing may be blocked. Remove the sample needle (see Section 7, paragraph 7.2.2) and use a syringe to force solvent through the needle or tubing to check for the blockage. Replace the needle or tube if the blockage cannot be cleared.

   If solvent does flow freely out the loose end of the PTFE tube, then proceed further:

4. Reconnect the needle to valve tubing.

5. Disconnect the PTFE tubing between the valve and the dispenser pump at the injection valve port 5.

6. Place a *capped* vial with solvent in the tray and program an injection.

7. Start the injection. Solvent should flow freely out of open valve port 5. If this is not the case, then the injection valve passage is blocked. Refer to Section 7, paragraph 7.3 for instructions on disassembling and rebuilding the Rheodyne 7010 injection valve.

If solvent does flow freely out port 5, then the tubing to the dispenser pump, or the dispenser pump tubing, or the waste tubing is blocked.

Leaks in the sample flow path may be isolated by applying a soapy solution to the various tubing connections and programming an injection of a newly capped empty vial. Bubbles will appear at the location of leaks.

## 6.6 TROUBLESHOOTING

Refer to Troubleshooting Table 6-1 for additional information on diagnosing and correcting problems with your instrument.



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| SYMPTOM | POSSIBLE CAUSE | |
|---|---|---|
| | Main power cord is not plugged in. | Plug in. |
| | Defective power supply electronics. | Contact Beckman Service. |
| | Incorrect value entered for current parameter. | Refer to Section 3 of this manual. |
| | Counter block of dispenser pump is not tightened. | Refer to Section 5, paragraph 5.2. |
| | Vials do not contain sufficient sample. | Refer to, Section 2, paragraph 2.9 of this manual for sample consumption with each method. |
| | Needle penetration depth is not properly adjusted. | Refer to Section 5, paragraph 5.3. |
| | Needle(s) damaged. | Refer to Section 7, paragraph 7.2. |
| | Blockage or leakage in the sample flow path. | Refer to Section 6, paragraph 6.5. |
| | Counter block of dispenser pump is not tightened. | Refer to Section 5, paragraph 5.2.3. |
| | Air leakage through and around vial septa. | Use new septa, tighten screw cap vials, or adjust hand crimper. |
| | Dispenser not properly calibrated. | Refer to Section 5, paragraph 5.2. |
| | Blockage or leakage in the sample flow path. | Refer to Section 6, paragraph 6.5. |

**Table 6-1 Troubleshooting**



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# Section 7
# Maintenance/Repair

## 7.1 GENERAL INFORMATION

This section of the manual covers the procedures used in the maintenance and/or repair of various components used in the instrument.

## 7.2 REPLACEMENT - SAMPLE TRANSPORT PARTS

Procedures for replacing the sample needle and connection tubing, air needle, and dispenser pump tubing are covered in the following paragraphs 7.2.1 through 7.2.3.

### 7.2.1 Replacing Sample Needle And Connection Tubing

To replace the sample needle and/or needle connection tubing, proceed as follows:

1. Make sure instrument is turned off, then unplug power cord from rear of instrument.

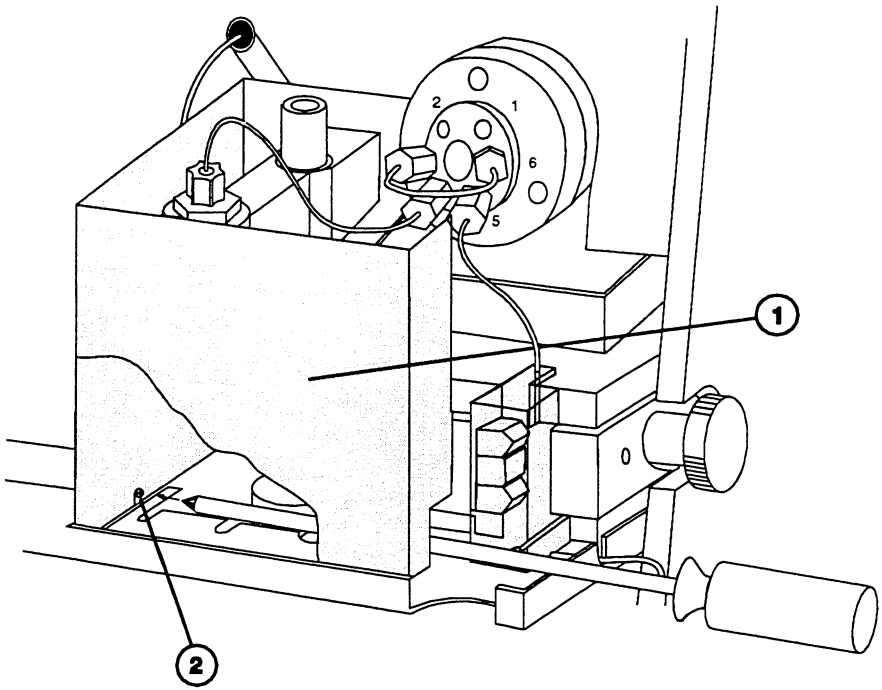
2. Disconnect air supply.

**⚠ WARNING**

**Be sure to disconnect the air supply to prevent personal injury! Failure to disconnect the air supply may result in accidental puncturing of your finger while working with the needle assembly if the needle assembly is activated.**

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3. Remove the injection system cover as shown in Figure 7-1.




1. Injection System Cover
2. Loosen screw with screwdriver then lift up and remove Injection System Cover.

**Figure 7-1 Removing Injection System Cover**



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4. Loosen the needle connection nut . Refer to Figure 7-2.

5. Move the needle arm down manually.

6. Remove the sample needle by pulling it out of its fitting, with the PTFE tubing attached to the needle.

7. Pull the sample needle out of the PTFE tubing and insert a new needle. The air seal shown in Figure 7-2 is a small slice of 1/16" tubing. Be sure to use the piece from the previous needle or cut a new piece. Also, the replacement needle may come with a white piece of plastic at the top end. This should be removed and discarded. Note the position of the nut and ferrule in Figure 7-2.

8. If the needle connection tubing needs replacement, loosen the nut that connects the tubing to the injection valve.

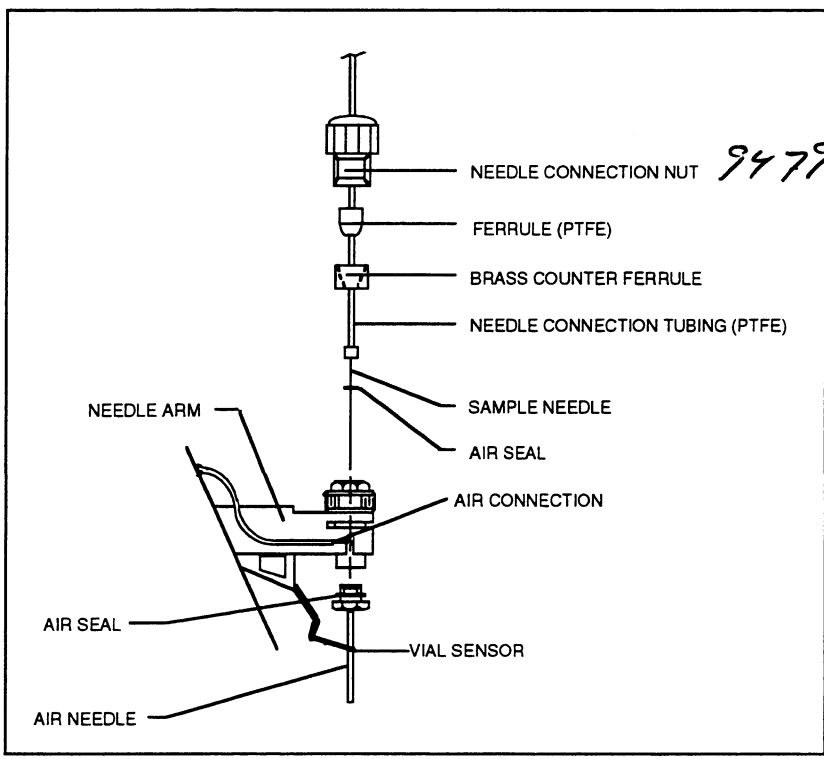



**Figure 7-2 Exploded Diagram of Needle Assembly**



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9. Connect the new tubing at the needle by sliding the end which has the larger inside diameter over the end of the needle. To get a better grip on the tubing, use a small piece of fine sandpaper or equivalent.

10. Reinstall the needle in its fitting and secure with the nut.

11. Move the needle arm upward.

12. Connect the loose end of the needle connection tubing to port 4 of the Rheodyne injection valve. Be sure to use a Rheodyne ferrule with a long male Rheodyne nut. Tighten only slightly past finger tight.

**⚠ CAUTION**

**Over tightening of the nut into the injection valve may cause the ferrule to close off the tubing.**

13. Check the needle penetration depth and adjust if necessary. Refer to paragraph 5.3.1

14. Reconnect power cord and air supply.

### 7.2.2 Replacement Of Air Needle

Referring to Figure 7-2, replace the air needle as follows:

1. Remove the sample needle as described in paragraph 7.2.1. Do not disconnect the PTFE tubing from the needle.

2. Unscrew the air needle and install the new one.

3. Reinstall the sample needle as described in paragraph 7.2.1.

### 7.2.3 Replacement Of Dispenser Pump Tubing

Referring to Figure 7-3, replace the dispenser pump tubing as follows:

1. Loosen the counter block of the dispenser by turning the knurled locking nut counter-clockwise.

2. Loosen the fitting that connects the PTFE inlet tubing of the dispenser to the Rheodyne injection valve.

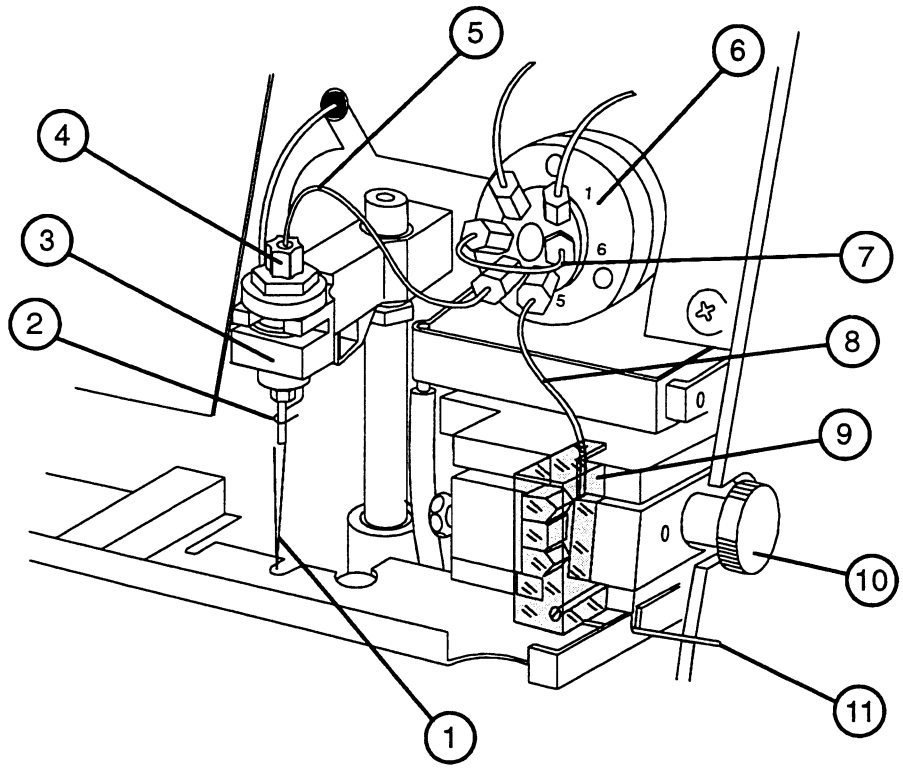
3. Remove the pump tubing cassette including the PTFE connection tubing by loosening the screw that attaches the cassette to the dispenser.



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4. Install the new pump tubing cassette.

5. Connect the PTFE tubing to the injection valve.




1. Needle Assy
2. Vial Sensor
3. Needle Arm
4. Needle Connection Nut
5. Needle Connection Tubing (PTFE)
6. Injection Valve
7. Sample Loop
8. Dispenser Inlet Tubing
9. Dispenser Pump Tubing Cassette
10. Locking Nut for Dispenser Counter Block
11. Drain

**Figure 7-3 View of Sample/Injection Systems**



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## 7.3 REPLACEMENT - INJECTION VALVE ROTOR SEAL

The rotor seal for the Rheodyne 7010 Injection Valve can easily be replaced by following the procedures outlined in paragraphs 7.3.1 and 7.3.2.

### 7.3.1 Removal Of Rotor Seal

Referring to Figure 7-4, remove rotor seal as follows:

1. Remove the three stator screws.

**NOTE**

**Do not change the setting of the three set screws in the stator.**

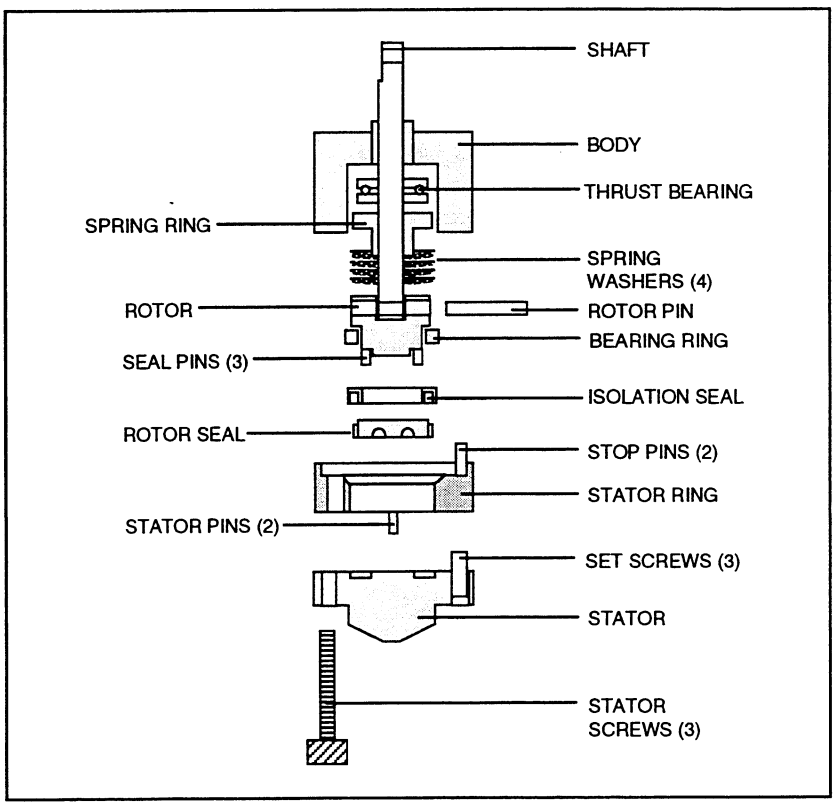



**Figure 7-4**
**Rheodyne Model 7010 Exploded View**



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2. Remove the stator and stator ring from the valve body by pulling axially to disengage the various pins.

3. To remove the rotor seal, pry it off of the four seal pins, using a screwdriver or knife blade.

4. The isolation seal and bearing ring usually are left in place because they rarely need changing.

### 7.3.2 Reassembly Of Injection Valve

Referring to Figure 7-5, install new rotor and reassemble injection valve as follows:

1. The rotor pin can be inserted in the rotor from one side only (the side without the peened closure marks around the pin hole). Do not try to force the rotor pin in from the wrong side. Be sure that the flat on the valve shaft faces away from the projecting end of the rotor pin.

2. Be sure that the rotor seal is correctly oriented as shown in Figure 7-5, with the rotor seal slots facing the stator and with the notch in the metal rim of the rotor seal in line with the rotor pin.

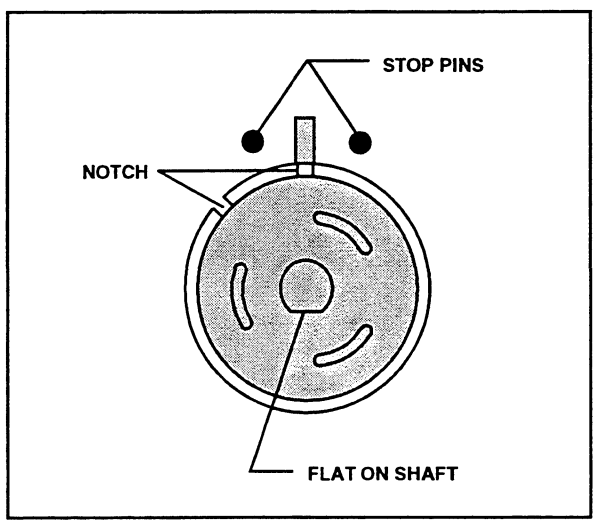



**Figure 7-5**
**Orientation of Rotor Seal Viewed From Stator**



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3. In replacing the stator ring, be sure that the two stop pins are replaced in their holes in the stator ring. Now push the stator ring squarely onto the rotor assembly so that the stop pins enter the mating holes in the body (with the rotor pin between the two stop pins) and the isolation seal slips inside the stator ring without hanging up.

4. Replace the stator by first pushing it onto the two pins on the stator ring and then adding the three stator screws. Tighten each screw a little at a time to keep the stator surface parallel to the stator ring surface. If the three set screws in the stator were left unchanged, then tighten the three stator screws until all parts are held firmly in place. The three set screws will ensure that the gap between the stator and stator ring is uniform and exactly as it was before disassembly.

5. If the set screws need adjusting because a new rotor seal was installed or because leakage has to be stopped, be sure that each set screw is turned an equal amount. This ensures that after the stator screws are retightened, the gap between the stator and stator ring is uniform.


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# Appendix A
# Specifications

The specifications for the 507 Autosampler are divided into eight catagories reflecting general, performance, as well as programming and options.

## *GENERAL*

**Sample Capacity**
Max. 97: 4 segments of 24 vials + satellite pos.

**Sample Identification**
Related to position in sample tray.

**Sample Vials**
Nominal - 2 mL vials; conical vials for small sample volumes.

**Power Requirements**
100, 120, 220, or 240 VAC; 50/60 Hz; 100 VA.

**Air Supply**
60 - 80 psi; CLEAN and DRY.

**Bench Space**
Width: 43 cm (16.93 in)
Depth: 51 cm (20.08 in)
Height: 30 cm (11.81 in)

## *PERFORMANCE*

**Reproducibility** (%RSD)
Flushed loop injection: better than 0.5 %.
Partial loop fill: better than 1.0 % .
µL pickup injection: better than 1.5 % (± 0.05 µL absolute).

**Cross-contamination** (1 µL blank inj. after sample inj.)
Max. flush volume (99 µL): < 0.2 %.
30 µL flush: < 0.5 %.
Needle wash (at satellite position): 0.0 %.

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**Minimum sample** (consumption for n injections per vial)
Flushed loop injection:
flush volume + n (1.5 x loop volume) + $(n-1)\left(\frac{\text{flush volume}}{2}\right)$

Partial loop fill:
flush volume + n (injection volume) + $(n-1)\left(\frac{\text{flush volume}}{2}\right)$

µL pick up injection: n (injection volume) µL

## *PROGRAMMABLE PARAMETERS*

**Methods - General** (battery backed up)
File number: regular methods (1 - 99)
µL pickup methods (100 - 109); not with RAC
event methods:110 - 125
Analysis time: 0 - 99 minutes, 59 seconds; 1 second increments
Loop volume: 1 - 999 µL in 1 µL increments

**Method - Regular**
Injection volume(s): max 9/vial; 0 - 999 µL in 1 µL increments.
Flush volume: 30 - 99 µL in 1 µL increments
Interval value (X): X = 1 - 96; after every X vials, the satellite vial is processed according to Method 99
Event Method number:110 - 125

**Method - RAC** (only)
Reaction time: 0 - 99 minutes, 59 seconds; 1 second increments

**Method - Event**
2-pos. stream switching valve (left side): 8 times
2-pos. stream switching valve (right side): 8 times
Auxiliary outputs 1 - 4: 4 times ON, 4 times OFF

## *SERIES*

Number of series: 1 - 99
Method number: 1 - 98, 100 -125
First/last vial: 1 - 96
Labelled vial output (for vial nr; max. 8 vials): 1 - 96


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## *PRIORITY*

Method number: 1 - 98, 100 - 125
Vial number: 1 - 97

## *ERROR DETECTION*

Sample transport; needle arm movement; dispenser function.

## *COMMUNICATION*

**Outputs**
Injection marker: normally open contact (floating); active low, open collector, TTL.
Next vial marker: active low, open collector, TTL.
Labelled vial: active high, open collector, TTL.
Stop: active low.
Vial number: 8-bit BCD, active high, TTL.
Auxiliaries 1-4 : active low, transistor, open collector.
Auxiliaries 1 and 2 inverted: active high, transistor, open collector.
Auxiliary power supply : + 24 V, 0.5 A.
Solvent selector valve: 4-bit BCD, true logic.

**Inputs**
Next injection: negative edge, pulled up.
Next vial: negative edge, pulled up.
Freeze: active low, pulled up.
Stop: active low.

## *OPTIONS*

- Integrated stream switching (ISS), including 2 high-pressure-port valves factory installed only.
- Integrated online reagent addition cassette (RAC).
- Sample tray cooling/heating, 4° to 60° C; controlled by an external refrigerated circulating bath.
- Column Heater.


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# Appendix B
# Stream Switching Valve Option

## B1.1 INTRODUCTION

The built-in Stream Switching Valve (SSV) option comprises two 6-port, 2-position rotary valves. These valves permit automated switching of columns to enhance analysis techniques. Some of the enhancements possible through SSV are as follows:

- Sample Preparation Time:
  Online chromatographic sample cleanup.

- HPLC Analysis Time:
  Increased sample throughput.
  Automated precolumn regeneration during analysis.

- Sensitivity:
  Trace enrichment.
  Sample cleanup (less electrode poisoning in Electro-Chemical Detector).

- Selectivity:
  Group separation (column switching).
  Chromatographic sample cleanup.

- Column Lifetime:
  Guard function of precolumn with automated regeneration.

The SSV is a factory installed option only.


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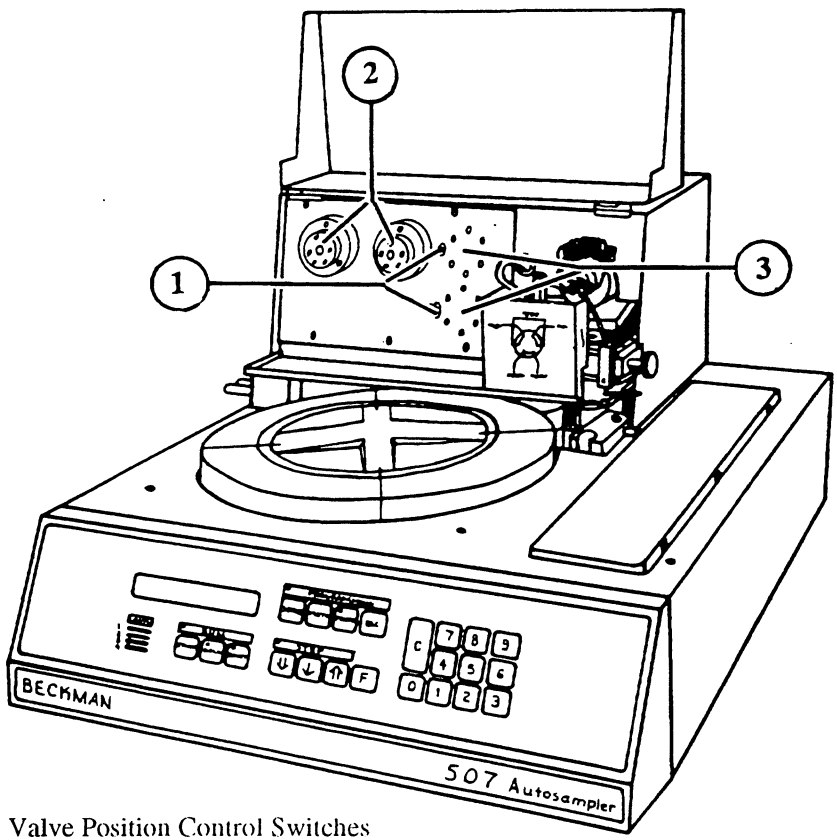
## B1.2 SSV OPTION DESCRIPTION

The SSV option is installed on the left side of the injection assembly. Figure B-1 illustrates the main components of the SSV.

As illustrated in Figure B-2, the valve positions are graphically illustrated via LEDs. The LEDs indicate which valve ports are internally connected.

Next to the LEDs are the two valve position switches. The upper switch manually controls the left valve and the bottom switch the right valve.

Each switch has three positions: left, center, right. The left position is Auto.




1. Valve Position Control Switches
2. Switching Valves
3. LED indicator shows valve position

**Figure B-1 Location of SSV Components**



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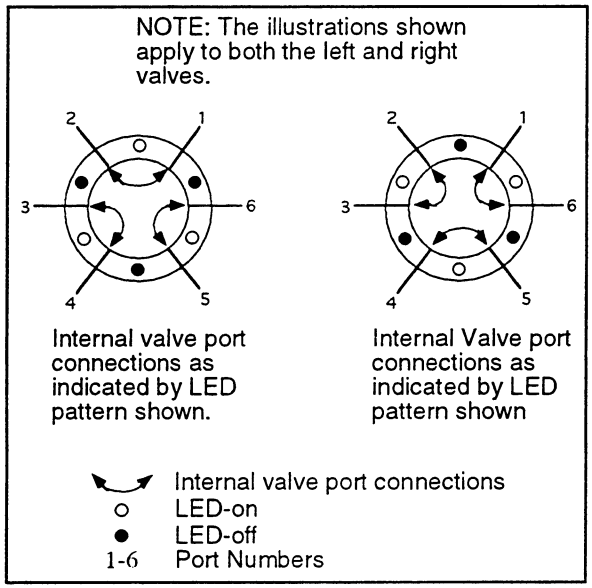



**Figure B-2 Valve Switching Diagram**

The switches must be in the left position when automatic operation of the valves using timed events from the Method is desired. The center position places the valve in position 1 which internally links port 1 to 2, 3 to 4, and 5 to 6. The right position places the valve in position 2 which internally links port 1 to 6, 2 to 3, and 4 to 5.

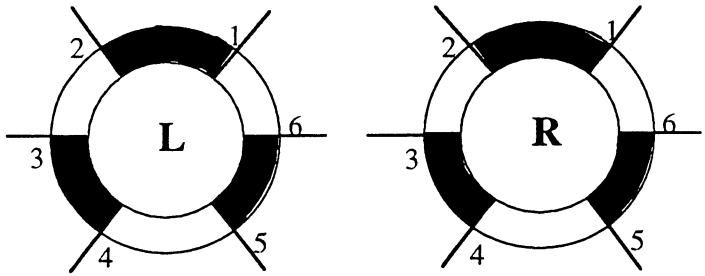
## B1.3 INSTALLATION

The valves are installed and checked-out at the factory before the instrument is shipped. An SSV option accessory kit is shipped with the SSV option. The kit includes the following items:

| Qty | P/N | Description |
|---|---|---|
| 2 | | Rheodyne parts kits |
| 2 | 243991 | Standard bushing |
| 2 | 243993 | Long bushing |
| 4 | 243992 | Ferrule |
| 1 | | Hex wrench, 5/64 in. |
| 1 | | Hex wrench, 9/64 in. |
| 10 | 890550 | SSV diagram panel charts |



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☐ ON POSITION
FLOW PATH:
2-3,4-5,6-1

■ OFF POSITION
FLOW PATH:
1-2,3-4,5-6

| Left Valve (L) flow pattern | Time min:sec | Remark |
|---|---|---|
| ■ | 00:00 | |
| ☐ | ... : ... | |
| ■ | ... : ... | |
| ☐ | ... : ... | |
| ■ | ... : ... | |
| ☐ | ... : ... | |
| ■ | ... : ... | |
| ☐ | ... : ... | |

| Right Valve (R) flow pattern | Time min:sec | Remark |
|---|---|---|
| ■ | 00:00 | |
| ☐ | ... : ... | |
| ■ | ... : ... | |
| ☐ | ... : ... | |
| ■ | ... : ... | |
| ☐ | ... : ... | |
| ■ | ... : ... | |
| ☐ | ... : ... | |

**Figure B-3 Flow Path/Time Diagram Chart**



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Before connecting any piece of tubing to the valves, make sure you have an adequate drawing of the switching diagram that can serve as plumbing guide. Use the Flow Path/Time Diagram Chart shown in Figure B-3 for this purpose. This will ensure correct external valve port numbering and correct internal valve port connection.

After reviewing the examples, copy the Flow Path/Time Diagram Chart and use to draw out your diagram. Use the items provided in the SSV option accessory kit and any other items, e.g., columns, etc., required along with the diagram to complete plumbing arrangements. The examples provided will also be used to plan timed events programming during operation of the instrument.

The initial position of the valves on power up is dependent on the setting of the switch next to the LEDs. These switches, one for each valve, can be used to set the initial position of each valve. Toggle switch left or right to set each valve's position. Position of each valve is indicated by the adjacent LEDs. Internal flow path is shown in Figure B-2. Note the position of each valve can be overriden via timed events programming (refer to paragraph B1.5).

## B1.4 COMMUNICATIONS

No additional electrical connections need to be made to control the valves. The electrical connections are factory installed when the valves are installed.

## B1.5 PROGRAMMING

Valve switching is programmed as an Event Method (Method numbers 110 through 125) as described in Table 3-4, Section 3 of this manual.

Note that the time base for Event Method programming begins at the moment of injection (start of analysis time).

Valves are not automatically reset to initial position prior to next analysis. This will have to be done either by using the valve position switches next to the LEDs or through the Event Method programming.

The programmed Event Method is incorporated in Regular Injection or µL Pickup Injection Method by entering the relevant Event Method number when programming the Method.


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## B1.6 OPERATION

All valves, including the injection valve, have been factory tested with IPA. Make sure that your HPLC solvent(s) are compatible with IPA. If not, start up with an intermediate solvent.

Step-by-step operational procedures are as described in Section 3 of this manual.

### *TIMING OF STREAM SWITCHING*

Switch timing of the valves is usually determined empirically with a manually operated HPLC system and for that purpose, both valves can be switched manually with the corresponding switches on the indication panel (make sure that air pressure is connected to instrument).

Use the switching diagram chart as shown in Figure B1-3 as a template to plan your valve switching arrangements. The chart can also be used as a template when programming the stream switching procedure in Event Method programming.

Refer to the diagram next to the LED indicators to get direct information on flow passages of your entire switching system.

### *AUTOMATIC SAMPLE PROCESSING WITH STREAM SWITCHING*

The valve position switches must be set to Auto (left most position) before pressing START. Automatic operation of the valves can always be overruled manually, by using the valve position switch adjacent to the LED indicators.

## B1.7 DIAGRAM CHART EXAMPLE

Refer to Figure B-4 for an example using the SSV in a timed switching arrangement for a protein purification application.

Autosampler wash and flush volumes, valve plumbing, and switching times are shown in Figure B-4. The right and left valve switching times as related to the chromatogram are also shown.

This application uses the Beckman Model 507 Autosampler with the Beckman Models 126 Pump and 167 Scanning Detector.


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# Appendix C
# Reagent Addition Cassette Option

## C1.1 INTRODUCTION

With the Reagent Addition Cassette (RAC) option installed, the 507 Auto-sampler automatically permits on-line mixing of a reagent in a constant ratio with the sample. This option is useful for internal standard addition or for precolumn derivatization. This option will be referred to as RAC on the instrument Display on power up.

Any "single step" derivatization can be processed automatically with a highly reproducible reagent/sample (1:1) mixing ratio and reaction time.

All three injections methods described in Section 3 of this manual can be used (flushed loop, partial loop filling and µL Pickup injection) with a few alterations in flush volumes and transport volumes as will be described later on in the programming section.

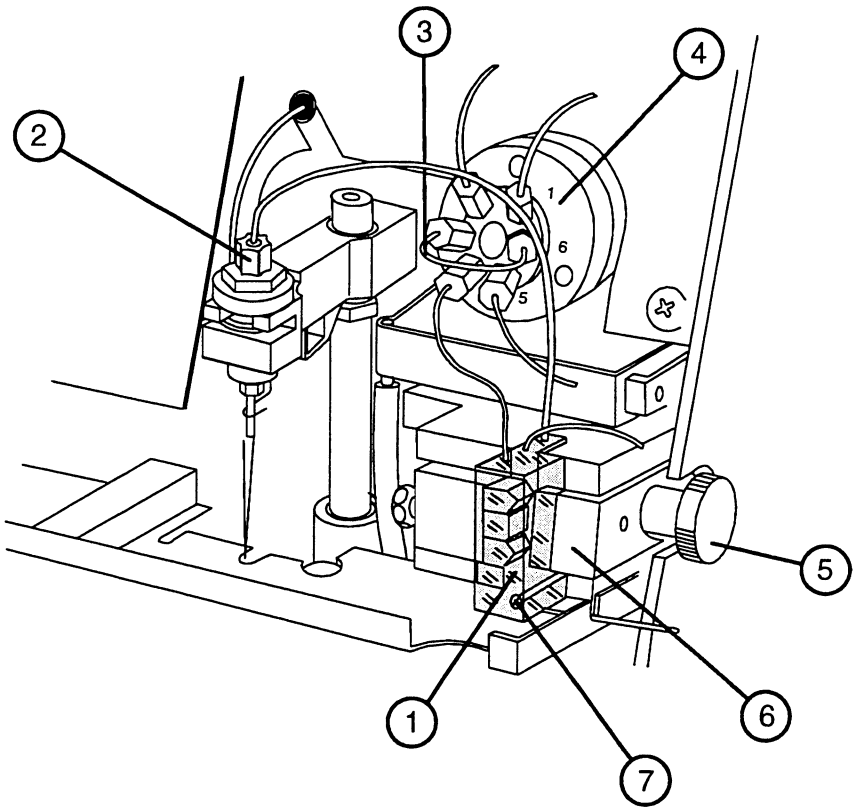
The RAC version of the instrument can be easily changed into a standard instrument. Only a few connections need to be exchanged when switching from RAC mode to STD (Standard) mode, or vice versa. The choice of either RAC or STD mode is asked for via the Display directly after power is turned on. The mode selected can be changed only after power switch is turned off and then on.

All materials in contact with sample, reagent or mobile phase are chemically inert including the pump tubing. The dual pump tubing cassette in the dispenser will mix reagent and sample in a 1:1 ratio.

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## C1.2 RAC OPTION DESCRIPTION

The RAC option is installed and becomes part of the injection assembly. The main components of the RAC option are illustrated in Figure C-1.




1. Dual Cassette Pump Cassette with Integrated Mixing Tee
2. Needle Connection Nut
3. Sample Loop
4. Injection Valve
5. Counter Block Locking Nut
6. Dispenser Counter Block
7. Cassette Mounting Screw

**Figure C-1 Location of RAC Components**



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## *LOADING AND DERIVATIZATION OF SAMPLE*

With Injection valve in load/react position (see Figure C-2), the Digital Dispenser System (DDS) will pump reagent and sample simultaneously into the "Y-junction" integrated in the dual pump cassette. The sample/reagent mixture is directed through the sample loop to waste. When the programmed amount of sample has entered the loop the dispenser stops. During the subsequent "reaction time" the sample/reagent mixture is allowed to react.

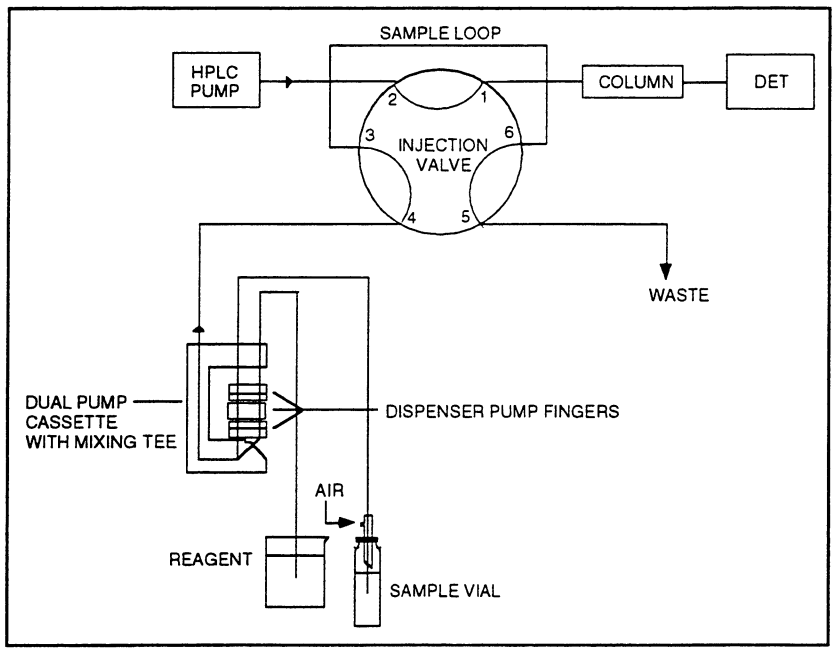



**Figure C-2**
**RAC Flow Diagram with Injection Valve Shown in Load Position**



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*INJECTION OF SAMPLE*

After completion of the reaction time, the injection valve is switched into inject position (see Figure C-3) to inject the derivatized sample onto the column.

After completion of the analysis time, the injection valve switches back to load position; the next sample can be processed.

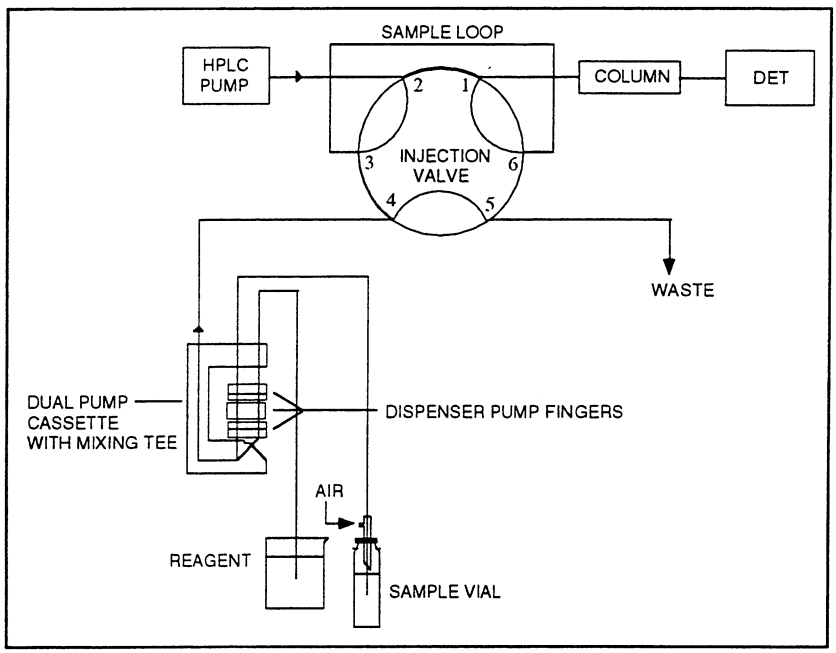



**Figure C-3 RAC Flow Diagram with Injection Valve Shown in Inject Position**



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## C1.3 INSTALLATION

The RAC option can be installed by the customer. The items provided in accessory kit P/N 240625 include everything needed to install the RAC option. The items included in the kit are as follows:

| Qty. | P/N | Description |
|---|---|---|
| 1 | 890551 | Dual cassette and replacement ferrule |
| 2 | 243992 | Ferrule, Rheodyne |
| 2 | 243993 | Long bushing, Rheodyne |
| 1 | 890552 | Silicone sleeve, 20 mm long |
| 1 | 890553 | PTFE tubing, 0.7 ID 300mm long, valve to waste |
| 1 | 947119 | 200 µL loop with ferrules and bushings |

Installation procedures to change from STD to RAC and vice versa are described in the following paragraphs. In both cases, you must remove the protective cover surrounding the injection assembly as shown in Section 7, Figure 7-1 to gain access to the DDS (Digital Dispenser System). Be sure to reinstall protective cover when you complete the following procedures.

### *CHANGING FROM STD TO RAC CONFIGURATION*

Referring to Figures C-4 and C-5, proceed as follows:

**⚠ WARNING**

**Disconnect the air supply to prevent personal injury! Failure to disconnect the air supply may result in accidental puncturing of your finger while working in the area of the needle assembly if the needle assembly is activated.**

1. Disconnect fittings at ports 4 and 5 of injection valve (needle and DDS connections, respectively).

2. Loosen cassette mounting screw. See Figure C-4.

3. Loosen counterblock locking nut. See Figure C-4.

4. Remove single pump tubing cassette. The waste tube must be disconnected from the single pump tubing cassette.



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5. Mount the dual pump tubing cassette but do not tighten the counter block locking nut.

6. Loosen the needle connection nut and remove both the needle and the connecting tube (lease needle attached to the tubing).

7. Remove the tubing from the needle by pulling them apart. Note the orientation of and remove the plastic nut, plastic ferrule, and brass ferrule seat from the tube.

8. Connect the middle tubing line from the dual cassette to the needle using the plastic male nut, ferrule and ferrule seat. Note that the end of the tubing is drilled out so that it will slip over the end of the needle. Be sure that the vial sensor is still in place around the needle.

9. Connect cassette and waste tubing as shown in Figure C-5. A small piece of silicon rubber tubing is provided with The RAC option for coupling the waste lines.

10. Install and tighten the cassette mounting screw. Do not tighten the counterblock locking nut yet.

11. Place a newly capped vial with pure solvent (water or methanol) in the tray.

12. Program an injection out of the vial with a long cycle time and more than one injection from the vial.

13. Start the injection. As soon as the needle pair penetrates the septum of the vial, solvent shold flow out the reagent line of the cassette. It will continue to flow after the DDS stops pumping because the vial is pressurized.

14. When the DDS stops pumping, tighten the counterblock locking nut until solvent just stops flowing out the reagent line.

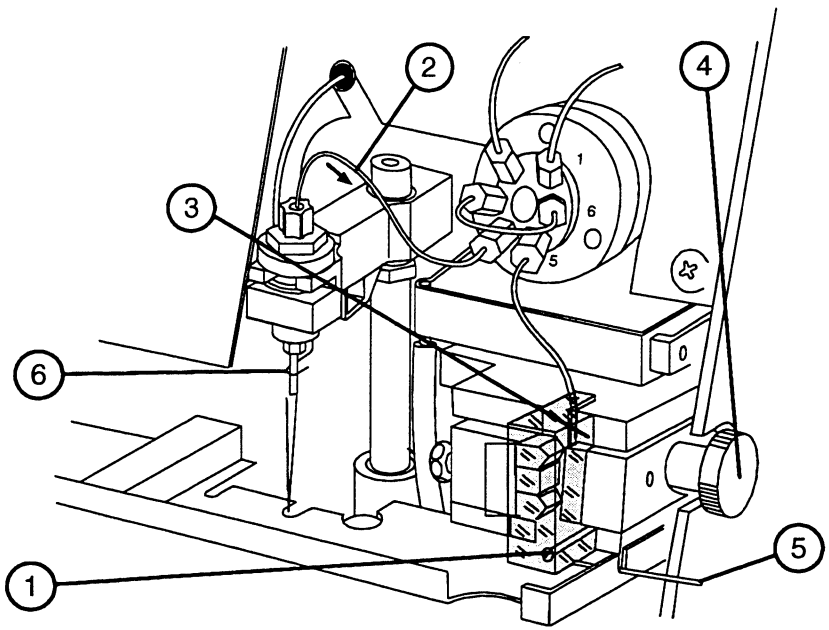
15. About the injection cycle and insert another newly capped vial in the tray.

16. Insert the reagent line of the cassette into a container of pure colvent (water or methanol).


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17. Make another injection and verify that reagent travels towards the cassette through the reagent line. Do this by briefly removing the line from the reagent to introduce an air bubble. If not, tighten the counter block locking nut slightly. If reagent still does not flow, the cassette may have air in the tubing. Loosen the counterblock until the lines fill and go back to step (14).

18. Operate the Autosampler for several injection cycles and check that the adjustment is still correct before running real samples. It may need to be readjusted due to the break-in of the cassette tubing.




1. Cassette Mounting Screw
2. Sample Flow
3. Standard Pump Cassette
4. Counter Block Locking Nut
5. Waste
6. Vial Sensor

**Figure C-4**
**Standard Plumbing Arrangement for Single Pump Cassette**


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*CHANGING FROM RAC TO STD CONFIGURATION*

Referring to Figures C-4 and C-5, proceed as follows:

1. Disconnect fittings at ports 4 and 5 of injection valve (pump tubing cassette and waste, respectively). Loosen cassettes mounting screw.

2. Loosen counter block locking nut.

3. Loosen needle connection nut and remove both the needle and connecting tube. Disconnect the tubing and save it with the dual cassette.

4. Remove dual cassette and mount the standard cassette.

5. Connect standard needle tubing to needle arm and injection valve (port 4). Note that one end of the tube is drilled open so that it can be slipped over the end of the needle.

6. Connect cassette tubing to injection valve (port 5) and waste tubing to waste.

1. Sample Reagent Mixture
2. Sample
3. Waste
4. Reagent
5. Dual Pump Cassette

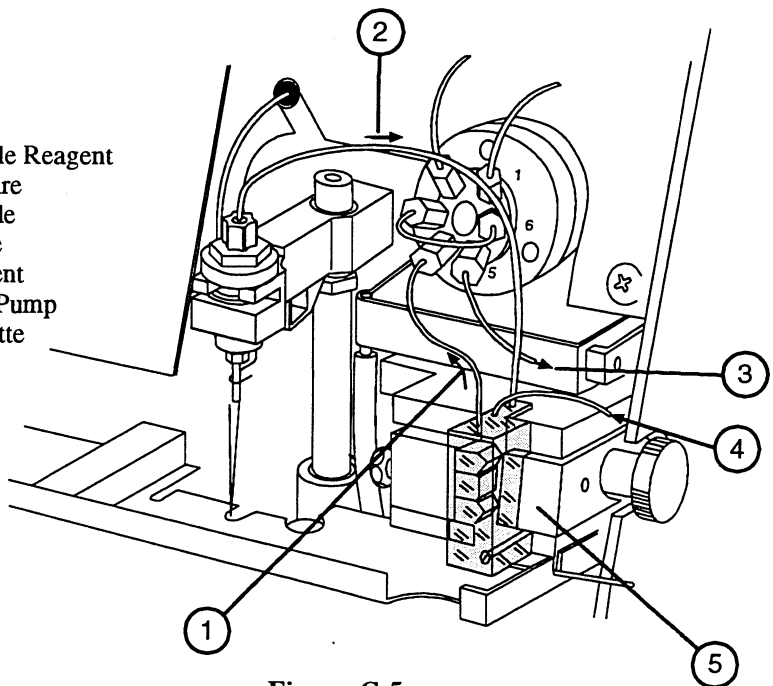



**Figure C-5**
**RAC Plumbing Arrangement for Dual Pump Cassette**



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## C1.4 SAMPLE CONSUMPTION

### *FLUSH VOLUME*

The dual cassette for RAC is placed between sample needle and injection valve thereby increasing the volume of the needle valve connection.

For Partial Loop Filling and Flushed Loop injections, a larger volume of sample is therefore needed for flushing to remove the previous sample quantitatively prior to filling the sample loop. The minimum (default) value for the flush volume in the RAC mode is therefore set to 50 µL (as compared to 30 µL in STD mode). When a sample loss of 50 µL is not acceptable, you can use the µL Pickup injection mode (Method 100 - 109).

### *INJECTION VOLUME AND LOOP VOLUME*

An equal volume of reagent is mixed with the sample volume before it enters the loop. The minimum loop size for Partial Loop filling with RAC is therefore twice as large as for STD. For STD, the minimum loop volume is 1.5 x injection volume; for RAC the minimum loop volume is 3 x injection volume.

For Flushed Loop injections the situation is the reverse. To fill the loop quantitatively it must be flushed with 1.5 x its volume. For RAC, only 0.75 x loop volume of sample is required since an equal volume of reagent is added. Therefore, the sample volume taken for Flushed Loop injection equals the loop volume (instead of 1.5 x loop volume as is for STD).

Therefore, with RAC the sample loss for Flushed Loop filling is not higher than for Partial Loop filling. Flushed Loop injection results in better reproducibility and is therefore recommended especially for RAC.

For µL Pickup injection the situation is more complex. Just keep in mind that the loop volume must be at least: 3 x injection volume.

## C1.5 PROGRAMMING

The RAC is programmed as described in Section 3 of this manual with the "React Time" parameter added in the METHOD program and selecting different values for minimum flush and loop volumes.

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| RAC Program Method Parameters | Valid Values |
|---|---|
| Method Number | 1 - 125 |
| Analysis Time | 0 - 99 min/ 59 sec. |
| Event Method Number | 110 - 125 |
| Loop Volume | Max. 999 µL |
| 1st Inj. Vol.- 9 th Inj. Vol. | 0 - 999 µL |
| Flush Volume | 50 - 99 µL |
| React Time | 0 - 99 min./59 sec. |
| Interval Value | 0 - 96 |

## C1.6 OPERATION

Review paragraphs C1.5 and C1.6 before proceeding with operation of the instrument. To operate the RAC equipped instrument, proceed as follows:

1. Turn on power and air supply if they are not already on. Display shows: STD (0) RAC (1).

2. Select RAC mode by pressing 1 on keyboard. Display shows: READY (X.X) RAC MODE. The X.X indicates software version.

3. Position reagent and waste container underneath reagent and waste tubing, respectively.

4. Ensure that vials contain sufficient sample, then place vials in the tray (caps cannot be twisted by hand when properly tightened).

5. Program analysis method(s). Refer to Section 3 of this manual for specific programming procedures.

6. Program SERIES and press ESC key. Refer to Section 3 of this manual for specific programming procedures.

Instrument is now READY for automatic processing of samples. Display shows: READY (X.X) RAC MODE.



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7. Press START key to initiate automatic processing.

8. Instrument automatically initiates READY mode after finishing the RUN program.

To interrupt automatic processing by HOLD, RESET or PRIORITY, refer to Section 3 of this manual.

## *USING THE SATELLITE POSITION*

The frequency of using the satellite position in sample processing is determined by the INTERVAL value in the Method program used for processing: INTERVAL value = 1 means that the satellite position is used after every sample vial.

What happens at the satellite position can be programmed in METHOD 99 only! (satellite method)

The satellite position can used for various purposes; Needle wash and external standard are described below as its most important applications.

## *NEEDLE WASH*

Place a vial with washing solvent at satellite position. Needle wash after every sample vial is programmed as follows:

| | | |
|---|---|---|
| Satellite Method: | METHOD NUMBER | 99 |
| | ANALYSIS TIME | 00/00 |
| | EVENT METH.NR | 000 |
| | LOOP VOLUME | Enter Relevant Value |
| | 1ST INJ.VOL. | 00 |
| Needle Wash: | FLUSH VOLUME | 99 µL for < 40 Vials; 50 µL for 50 to 96 vials |
| | REACTION TIME | 00/00 |
| Injection Method: | Enter relevant values for sample injection; enter **1** for INTERVAL value. | |



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*EXTERNAL STANDARD*

Place a vial with standard at satellite position. Injection of standard after every "x" number of samples is programmed as follows:

Satellite Method: METHOD NUMBER 99

Enter relevant values for next parameters to program standard injection from the Satellite vial.

Regular Method: Enter relevant values for sample injection; enter "x" for INTERVAL value.

*AFTER USE*

If instrument is not to be used within a day after the last analysis, replace reagent with a pure (cleaning) solvent; then, program an injection using the pure solvent to clean sample, reagent, and waste tubings.

## C1.7 TROUBLESHOOTING

The following Troubleshooting Table C-1 should be regarded as additional to the Troubleshooting Table 6-1, Section 6, of this manual.



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| SYMPTOM | POSSIBLE CAUSE | REMEDY |
|---|---|---|
| a. No sample is injected (with undamaged needle) | Needle penetration depth not properly adjusted. | Refer to paragraph 5.3. |
| | Not enough sample in vial. | Refer to paragraph 2.9. |
| | Reagent container empty. | Refill. |
| | Leaking connection(s). | Locate and correct. |
| | Block in liquid flow path. | Refer to paragraph 6.5. |
| b. Bad reproducibility | Derivatization process not reproducible. | Optimize reaction. |
| | Block in liquid flow path. | Refer to paragraph 6.5. |
| | Leakage in liquid flow path. | Locate and correct. |
| c. High cross contamination | Block in liquid flow path. | Refer to paragraph 6.5. |
| | Leakage in liquid flow path. | Locate and correct. |

**Table C-1 Troubleshooting RAC System**

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## *TRACING BLOCKAGE/RESTRICTION IN THE LIQUID FLOW PATH*

There are two areas of potential blockage and/or restriction: high pressure, which includes the HPLC Pump, injection valve, column/detector and interconnecting tubing; low pressure which includes the injection valve assembly, reagent plumbing, sample plumbing, and dual pump cassette.

Referring to Figure C-2 flow diagram, the high pressure system can be checked out by observing solvent flow from each component in the system in sequence, e.g., with HPLC pump set to 1 mL/min, check flow at port 2 of injection valve, followed by checking port 1, and leading to column/detector and interconnecting tubing. You will need to disconnect and then reconnect each component as you move throughout the high pressure system.

If any blockage or restriction is found, correct problem before proceeding with operation of instrument.

Checking out the low pressure system can be accomplished by weighing a sample vial filled with HPLC grade water before and after a preset injection volume, e.g., 20 µL, is made. The reduction in weight should equal the reduction in volume, i.e., one µg of water equals one µL of water. This method will confirm proper operation of sample flow and also accuracy of pumping system.

Since the pumping system pumps equal amounts of liquid from the sample vial and the reagent bottle (see Figure C-2 for flow diagram), twice the volume of liquid will appear at the waste line. If volume is not doubled or you do not observe a reduction of volume from the reagent bottle after each injection or flush, check dual pump cassette and associated tubing for restriction and/or blockage. Remove dual pump cassette and clean or replace as necessary. Also make sure no air bubbles are trapped in lines. Purge air bubbles if they are present.



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# APPENDIX D
# SPARE PARTS

*Spare Parts List*

| Beckman Part No. | Description |
|---|---|
| 947971 | Air Filter |
| 947978 | Seal Ring M5; Washer for Air Needle |
| 947986 | Air L-Connection T6 - T6 |
| 873155 | Power Cord, USA |
| 890518 | Air Needle |
| 890519 | Sample Needle |
| 887954 | Vials, 2 mL screw-cap; Pkg. of 100 (Sun Brokers #1260A) |
| 243992 | Ferrule, Rheodyne |
| 890531 | Air Tubing OD 6 mm; Connects inlet air to filter |
| 947938 | Conical Ring; Ferrule Seat |
| 947937 | Male Nut M6; For Sample Tube |
| 947934 | PTFE Ferrule, 1/16 inch |
| 243993 | Long Male Nut, Rheodyne |
| 947945 | PTFE Tubing, 1/16 inch by 0.3 mm; Sample Needle to valve including ferrule |
| 947061 | Pump Tubing Cassette Assembly |
| 243649 | Rheodyne 7010 Rotor |
| 241569 | Wash Reservoir, 30 mL |

*Options*

| | |
|---|---|
| 240625 | Reagent Addition Cassette |
| 240629 | Sample Tray Heat Exchanger |



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