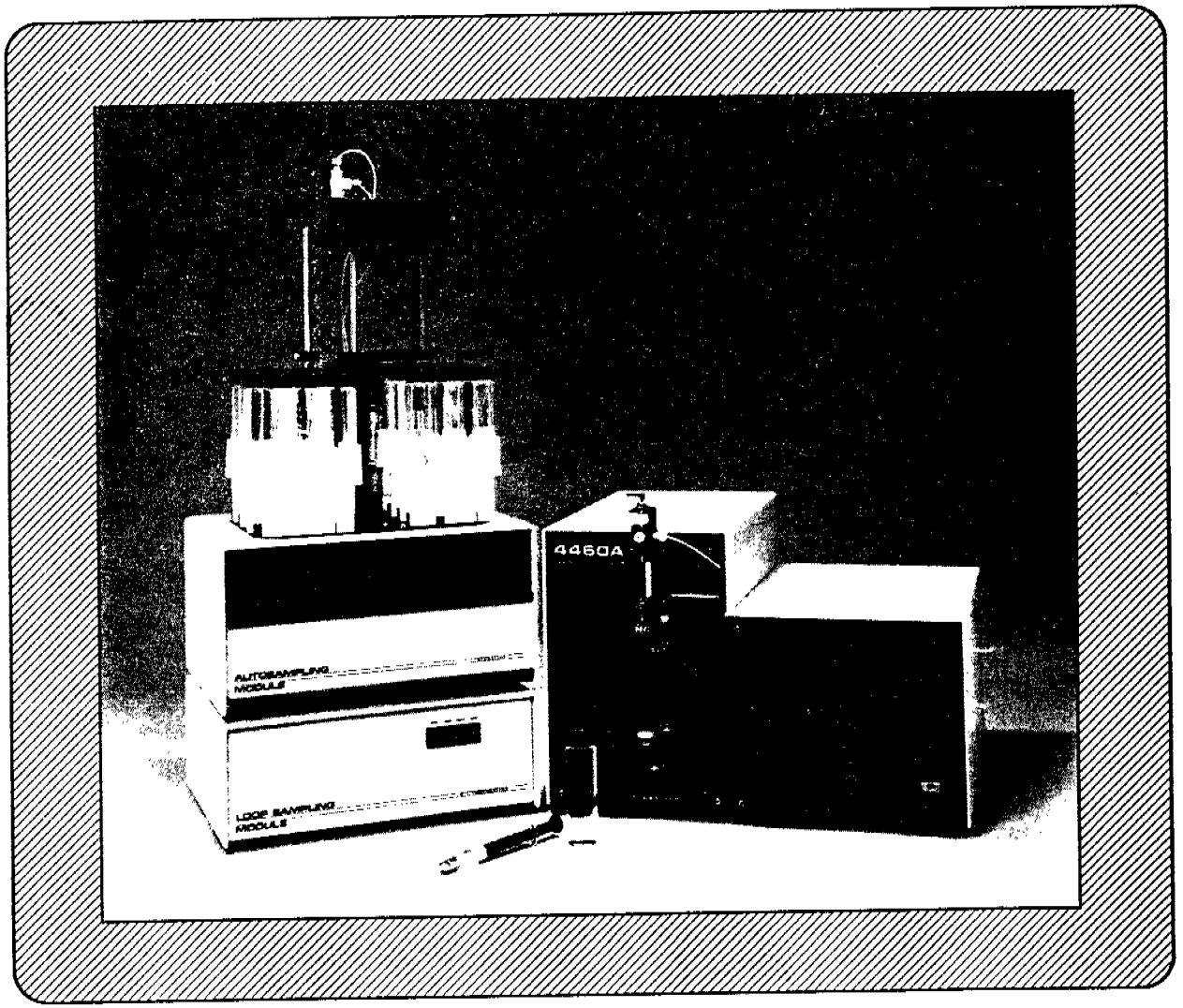
4460A Purge and Trap Concentrator

Operating and Service Procedures



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Limited Warranty

O.I. Corporation warrants each Model 4460A Purge and Trap Sample Concentrator and its optional equipment against defects in materials and workmanship under normal use and service for a period of ninety (90) days. Equipment installed by O.I. Corporation is warranted from the installation date, all other from the ship date. If purchaser schedules or delays installation more than 90 days after delivery, then warranty period starts on the 91st day from date of shipment. This warranty extends only to the original purchaser. O.I. Corporation will, at its option, repair or replace equipment that proves to be defective during the warranty period, provided the equipment is returned to O.I. Corporation at the expense of the purchaser. Parts, labor and return shipment to the customer shall be at the expense of O.I. Corporation. Parts used and labor performed during on-site warranty service requested by the purchaser shall be at the expense of O.I. Corproation. Travel costs shall be at the expense of the purchaser.

As a condition of warranty, the purchaser shall regularly maintain the equipment as specified in the Model 4460A Purge and Trap Sample Concentrator Operating and Service Manual provided with the equipment. Evidence of such maintenance in the form of a maintenance log book is recommended. Expendable components which are to be replaced regularly according to the manual are not covered under this warranty.

THE FOREGOING WARRANTY IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESS OR IMPLIED, INCLUDING BUT NOT LIMITED TO ANY WARRANTY OR MERCHANTABILITY, FITNESS OR ADEQUACY FOR ANY PARTICULAR PURPOSE OR USE. O.I. CORPORATION SHALL NOT BE LIABLE FOR ANY SPECIAL, INCIDENTAL, OR CONSEQUENTIAL DAMAGES, WHETHER IN CONTRACT, TORT, OR OTHERWISE.

Any service requests or questions should be directed to the Customer Service Department 409-690-1711.

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Chapter 1
Model 4460A
Sample Concentrator

The Model 4460A Sample Concentrator is designed to (1) strip purgeable organic compounds from a water sample, (2) concentrate them on a specified sequence of adsorbent materials, and (3) rapidly desorb them using heat and carrier gas flow onto a gas chromatographic column. The 4460A should be regarded as one component of an analytical system for purge and trap analysis. Other necessary components include a gas chromatograph (GC), a 4460A/GC interface kit, single or multiple detectors, and a data handling device for quantification of detector signals. An autosampler for introducing water samples to the concentrator may also be part of the overall analytical system.

The 4460A consists of a vessel for purging organics from solution and a trap for concentrating the purged compounds, interfaced to each other and to the GC by a set of valves and fluid flow lines. By proper manipulation of valves and trap temperature through a specific time sequence, quantitative analysis of purgeable organics is achieved.

In order for you to understand the way the 4460A has been designed, some basic operational concepts, features, and specifications are outlined in this chapter.

Operational Concepts

Instrument STATES

The various sets of valve and trap temperature combinations defined for analysis using the 4460A are called **states.** The eleven primary states are called:

STANDBY	DESORB PREHEAT
PURGE READY	DESORB W/DRAIN
PURGE	DESORB W/O DRAIN
DRY PURGE	BACKFLUSH BAKE
PURGE COMPLETE	DRAIN
DESORB READY	

Other states have been defined for use with the optional autosampler system. These are:

LOOP FILL	INJECT RINSE 1
LOOP INJECT	DRAIN RINSE 1/LOOP RINSE 2
POSITION TO RINSE	INJECT RINSE 2
LOOP RINSE 1	DRAIN RINSE 2

The 4460A is taken through specific sequences of these states under control of its microprocessor. Various different analytical procedures for purgeable organics require different times and temperatures of these states, so the 4460A has been default-programmed to allow the setting of one of eight pre-defined arrangements called Method Designators. Each of these is set by a unique combination of DIP switches in the 4460A as described in **Chapter 2** under **Right Bay Components.**

Each unit is shipped from the factory set to Method Designator 601, which corresponds to the conditions of analysis specified by EPA Method 601, Purgeable Halocarbons.[1] When the instrument is powered up, the Method Designator, "601" in this case, is displayed for 10 seconds in the Temperature display to confirm which set of conditions were loaded into memory. This set of conditions will remain in memory until the Method Designator DIP switches are changed and the unit re-powered, or until the user manually changes any of the conditions.

[1] *Method 602 - Purgeable Aromatics, Federal Register, Vol. 49, No. 209, pages 40-48, Friday, October 26, 1984 This document is available from United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268.*

Sequencing Through STATES

When the 4460A is operating with its timer running, the microprocessor will advance the system through the following sequence of primary states, each existing for the time specified by the Method Designator, or for some states, until some other condition is met:

STATE	CRITERIA FOR ADVANCE PAST
STANDBY	Setpoint Temp Achieved
PURGE READY	External Signal Received
PURGE	Time Elapsed
DRY PURGE	Time Elapsed
PURGE COMPLETE	Setpoint Temp Achieved
DESORB READY	External Signal Received
DESORB PREHEAT	Setpoint Temp Achieved
DESORB W/DRAIN	Time Elapsed
BACKFLUSH BAKE	Time Elapsed

The states DRAIN and DESORB W/O DRAIN are not in the automatic sequence, but can be manually set.

Functions of each of these primary states, including flow diagrams and specific conditions for advance, are described in **Chapter 4.**

When the autosampler is enabled, several other states are added to the sequence, each lasting for a time specified by the Method Designator, unless changed manually. In this case, the sequence of states is:

STATE	TYPE
STANDBY	PRIMARY
PURGE READY	PRIMARY
LOOP FILL	AUTOSAMPLER
LOOP INJECT	AUTOSAMPLER
PURGE	PRIMARY
DRY PURGE	PRIMARY
PURGE COMPLETE	PRIMARY
DESORB READY	PRIMARY
DESORB PREHEAT	PRIMARY
DESORB W/DRAIN	PRIMARY
POSITION TO RINSE	AUTOSAMPLER
LOOP RINSE 1	AUTOSAMPLER

(cont'd)

STATE	TYPE
INJECT RINSE 1	AUTOSAMPLER
DRAIN RINSE 1/LOOP RINSE 2	AUTOSAMPLER
INJECT RINSE 2	AUTOSAMPLER
DRAIN RINSE 2	AUTOSAMPLER
BACKFLUSH BAKE	PRIMARY

Functions and flow diagrams of the autosampler states are also presented in **Chapter 4.**

Components Which Are Heated

Various components of the 4460A must be heated to meet proper analysis conditions for purgeable organics. Components whose temperatures are controlled by the microprocessor are:

Trap: Purge Temp	Valve Oven
Desorb Temp	Transfer Line
Bake Temp	External Heated Device

Actual and set point temperatures of each of these components can be displayed. In addition, the temperatures for Trap Preheat and for the GC column oven can be displayed.

Features

Sample Concentrator

- Fully complies with EPA purge-and-trap method protocols
- Exceeds performance requirements for EPA purge-and-trap method protocols
- Designed for use with wide-bore capillary and packed columns
- Provides ultra-low dead volumes from sample inlet to column transfer
- Microprocessor controlled, fully automatic, fully programmable
- Digital display of all actual set point, times and temperatures
- Default programmed for EPA Method 601/502.2 and all other current EPA methods
- Compact, lightweight, portable
- Simple to program and operate - easy to service
- Dry purge function is standard
- Back-flushed bakeout of trap is standard
- Can be used with 5 or 25 ml sparging vessels
- Both frit and needle spargers are available
- Power and temperature feedback for an external heated device (hot purge) is standard
- Heated valve oven and transfer line with temperature setpoints adjustable and actual temperatures displayed
- Trap heating from ambient to 180°C in 12 sec. with no overshoot
- GC quality flow controller, pressure regulator, and pressure gauge for purge gas flow control
- Low-dead-volume interface kits for popular GCs
- All sample wetted lines are fused-silica coated stainless steel (no Teflon)
- All sample flow pathway fittings are gold-plated
- Water management system for trap desorption is standard
- Carryover less then 0.3% for all 601/602 components

- GC interface allows new sample purge while previous sample is eluting from GC and automated analysis of multiple samples
- Low-dead-volume heated injection port allows sample injection directly onto capillary GC column
- Manual override of automatic operation - ability to single step through states
- Auto-run mode for repetitive analysis of blanks or sample re-purge, and for auto sampling
- Fully interchangeable with Tekmar LSC-2
- Fully compatible with Tekmar ALS
- Optional VOA vial autosampler with sample loop injection for drinking and waste water - optional Standards Injection Module for internal and surrogate standards
- Optional 16 and 32 station Multiple Purging Module for hazardous wastes and solids

VOA Vial Autosampler

- Comprised of a Loop Sampling Module and an Autosampling Module
- Capacity for 76 14-ml vials or 27 40-ml vials
- Standard EPA-approved VOA vials fit directly into sampler. No need for opening vial or manual sample transfer
- Septum piercing needle transfers sample automatically from closed vial to purge vessel via 5 ml or 25 ml sample loop
- Calibrated loop injection of sample maximizes accuracy and repeatability of sample volume, minimizes contamination
- Changeable sample loops allow different sample volumes
- Two automatic rinses of sample lines and purge vessel between samples minimize carryover
- Rinse station allows methanol or other solvent solution to be used for rinse to minimize carryover
- Immediate replicate sampling from vial is automatic to protect sample integrity
- Number of vials to be sampled is selectable
- Number of replicates to be sampled per vial is selectable
- Loop Sampling Module can be used for unattended process stream monitoring
- Septum piercing needle can be inserted manually into individual sample vials even without the Autosampling Module, for loop sampling from the vial without exposing the sample to the air
- Automatic injection of Internal and/or Surrogate Standards is optional

Specifications

Principle of Operation

Aqueous samples containing volatile organic compounds are purged with an inert gas for a fixed period of time. Volatile compounds are sparged from the sample and collected on a sorbent trap contained within the 4460A. The trap then is rapidly heated and the compounds collected are transferred as a "plug" under a backflush flow of inert gas to an external gas chromatograph. Conventional chromatographic techniques then are used to identify and quantify volatile sample components.

Physical Design

Dimensions

- 14.8 inches (380 mm) wide
- 10.4 inches (262 mm) high
- 11.1 inches (283 mm) deep

Sample transfer line

- Fused-silica-lined stainless steel (replaceable)
- 48 inches (122 cm) long
- 0.062 inches (1.6 mm) OD
- 0.020 inches (0.5 mm) ID

Weight - 30 lbs (13.6 kg)

Purge Gas Filter - 13X molecular sieve standard

Tubing and Valving - All gas flow lines are stainless steel. All lines in contact with sample are fused-silica-lined stainless steel. All fittings in contact with sample are gold-plated. Internal volume seen by sample downstream of purge vessel is heated and totals less than 100 microliters. Trap valve is 6-port stainless steel, pneumatically actuated, low-dead-volume.

Electrical Requirements

- 115/230 VAC, selectable by jumper
- 50 or 60 Hz, automatically compensated
- 800 W at 115 VAC

Gas Requirements

Purge Gas - Helium or Nitrogen, 99.998% or better purity recommended, regulated in the range of 30 to 130 psi (207-900 kPa). Must be compatible with GC detector.

Actuator Gas - Dry air or other dry non-combustable gas, house gas or commercial grade, regulated in the range of 40 to 80 psi (276-552 kPa) (purge gas can be used).

Carrier Gas - As required for GC and detector, but regulated at least in the range of 0-250 psi (0-1724 kPa) for the 4460A trap valve (typically uses same as used for purge gas).

Traps

All traps are packed according to methodology described in current EPA Methods.

Included:

- Two of OV-1/Tenax/Silica Gel
- One of OV-1/Tenax
- One empty

Available:

- Tenax only
- OV-1/Tenax/Silica Gel/Charcoal
- Custom upon request

Spargers

Included:

- One of 5 ml frit sparger (can be used for 10 ml samples)

Available:

- 25 ml frit sparger
- 5 and 25 ml needle spargers
- Amber frit and needle spargers

STATES of Operation *(state and trap status indicated by LED)*

By Automatic Sequence

- STANDBY
- PURGE READY
- PURGE
- DRY PURGE
- DESORB READY
- DESORB PREHEAT
- DESORB W/DRAIN
- BACKFLUSH BAKE

With Autosampler

- LOOP FILL
- LOOP INJECT
- POSITION TO RINSE
- LOOP RINSE 1
- INJECT RINSE 1
- DRAIN RINSE 1/LOOP RINSE 2
- INJECT RINSE 2
- DRAIN RINSE 2

By Manual Command

- DRAIN
- DESORB W/O DRAIN

Time Functions

General Performance

- 1 second increment digital setpoints
- 100% accuracy (tied to line frequency)
- Displayed in MM:SS format from 00:00 to 99:59
- Default values of setpoints set by Method Designator setting upon power up

Programmable Ranges - Primary States

- PURGE: 0 to 99 min: 59 sec.
- DRY PURGE: 0 to 99 min: 59 sec.
- DESORB: 0 to 99 min: 59 sec.
- BACKFLUSH BAKE: 0 to 99 min: 59 sec.

Programmable Ranges - Autosampler States

- LOOP FILL: 0 to 99 min: 59 sec.
- LOOP INJECT: 0 to 99 min: 59 sec.
- LOOP RINSE: automatically set to LOOP FILL + 20 sec.
- DRAIN RINSE: automatically set to LOOP INJECT + 15 sec.

Temperature Functions

General Performance

- 1°C increment digital setpoints
- 0.5°C full scale absolute accuracy
- 0.12°C repeatability
- Ambient temperature compensation
- Default values of setpoints set by Method Designator setting upon power up

Programmable Ranges - Trap

- Purge: 0° ambient to 250°C
- Desorb: 0° ambient to 250°C
- Bake: 0° ambient to 250°C

Programmable Ranges - Other Heated Components

- Valve Oven: 0° ambient to 250°C
- Transfer Line: 0° ambient to 150°C
- External Heater: 0° ambient to 300°C

Programmable Threshold Settings

- Column Temperature (advance to DESORB PREHEAT) - 0° to 300°C
- Desorb Preheat (advance to DESORB) - 0° to 300°C
- Advance to PURGE READY - Set at 2°C above Purge Temp Setpoint

Trap Heating and Cooling Rate

- Heating - 800°C/minute (25°C to 180°C within 12 seconds)
- Cooling - 300°C/minute (180°C to 30°C within 30 seconds)

Electrical Outputs

- Activates 2 sec. relay closure upon advance to, or end of, DESORB. DIP switch selectable, rated 120 V, 5 A max
- Activates 2 sec. relay closure upon advance to STANDBY including each time power is turned on, rated 120 V, 5 A max
- Activates optical couplers to provide same output information

Electrical Inputs

- Accepts (2 sec.) switch closure to advance from PURGE READY to PURGE
- Accepts (2 sec.) switch closure to advance from DESORB READY to DESORB PREHEAT
- Accepts optical coupler input to provide same input control
- Accepts Type K thermocouple input to control advance from PURGE

Autosampling Module

Nominal Line Voltage

- 100 ± 10 VAC, 117 ± 15 VAC,
- 234 ± 30 VAC, 50/60 Hz

Line Voltage Noise Tolerance

- ±170% of nominal line voltage, 10 microsecond pulses at any phase angle

Power Consumption

- 90 watts maximum

Ambient Temperature Range

- 0° to 40°C

Humidity

· Up to 100% relative humidity if connected to line voltage

Dimensions

Cabinet:

· Depth - 23 cm (8.9")
· Width - 29 cm (11.5")
· Height - 16 cm (6.0")

Overall With Wash Station:

· Depth - 26 cm (10")
· Width - 29 cm (11.5")
· Height - 32 cm (17")

Weight

· 10.4 kg (22 pounds)

Tube Capacity

· 114 - 10 mm to 13 mm tubes
· 76 - 16 mm tubes, 17 or 18 mm vials
· 27 - 28 mm tubes (40 ml EPA vials)

Loop Sampling Module

Nominal Line Voltage

· 115 VAC 60 Hz standard, specify other

Ambient Temperature Range

· 0° to 40° C

Dimensions

· Depth - 23 cm (9 in.)
· Width - 28.5 cm (11 1/4 in.)
· Height - 11.5 cm (4 1/2 in.)

Pump

· Type: Peristaltic metering
· Speed: 15 RPM
· Rate: 18 ml/min with standard tubing
· Tubing: Silicone rubber, 3/16 in. (4.75 mm) ID

Sample Loops

· Material: TFE Teflon-flared ends
· Volume:
  - 5 ml installed standard
  - 25 ml in Startup Kit
  - Other volumes available

Power Consumption

· 90 watts Maximum

Weight

· 5 kg (10 lbs)

Humidity

· Up to 100% relative humidity if connected to line voltage

Sample Valve

· Type: 6-port sampling logic
· Material: TFE Teflon wetted parts
· Porting: 1/4-28 threads
· Actuation: Dry air or nitrogen
· 40 to 80 psi (276 - 552 kPa)

Manual Controls

· Raise (for Autosampling Module)
· Lower (for Autosampling Module)
· Wash (for Autosampling Module)
· Pump (on/off)

Standards Injection Module

The Standards Injection Module (SIM) is used in conjunction with the 4460A Sample Concentrator and its VOA vial autosampling system. It consists of an electrically actuated injection valve with a calibrated 10 microliter bore and a motor driven syringe working together to repeatably inject 10 to 90 microliter volumes of internal and/or surrogate standard solution into a sample to be analyzed by purge and trap methods. It can be operated manually as a stand-alone module, but is designed to accept control signals from the 4460A for automated operation.

Injection

- Injection Volume: 10 µl to 90 µl in 10 µl steps
- Injection Volume Precision: ± 0.2%
- Volume Metering Device: Sampling valve
- Sampling Valve Actuator: Electric
- Syringe Motive Force: Electric drive lead screw
- Syringe Travel Resolution: 0.0005 inch

Loading

Standards reservoir syringe may be power-filled using three-speed motor with syringe in place or by manually replacing empty syringe.

Mode

- Automatic: Automatic standard injection(s) 3 sec. after beginning of sample injection
- Manual: Injects when operator pushes "MANUAL INJECT" button

Materials

- Valve: Kel-F body, TFE rotor
- Hamilton #1010-TFE LL
- Sample Reservoir: Gas tight syringe,
- Fluid Path: All internal lines stainless steel

Dimensions

- 2.8 inches high
- 8 inches wide
- 11 inches deep
- 4 lbs

Electrical Requirements

- Power Supply: External, + 12 volts DC ± 5% at 2 amps
- Signal Input: External dry contact closure, 12 volt active
- Low pulse, or 5 volts TTL active low pulse (50 ms minimum pulse width)

Controls

- "ON" position:
  - Enables the unit to perform automatic injections under external control
- "BYPASS" position:
  - Disables automatic injection
  - Allows manual injection
  - Allows manual syringe operation for filling or priming system

All electrical requirements are supplied by 4460A.

- Manual Inject: Immediately initiates an injection cycle of the total injection volume
- Syringe Advance: Moves syringe plunger forward to prime system or drain syringe
- Syringe Fill: Moves syringe plunger to fill syringe or returns syringe to starting position
- Injection Multiplier: Settable from 1 to 10 injections (10 µl to 90 µl) to adjust injection volume

Additional Useful Hardware

There are several kits available for use with the 4460A. A Startup Kit is included with each unit, and GC Interface kits, Hookup Kits, and Syringe Kits may be purchased separately.

Startup Kit *(OIC Part #177-618)*

This kit is included with the 4460A. It contains all materials necessary for the operation of the 4460A except for gas lines (in Hookup Kit) and the GC interface. Included in the Startup Kit are a heated transfer line, syringes, septa, and spare traps.

Hookup Kit *(OIC Part #177-592)*

This kit includes tubing and fittings for connecting the 4460A to purge and actuator gases. It includes 5 feet of 1/8 inch OD copper tubing, 1 foot of 1/8 inch OD Teflon tubing, one tee fitting and one tube adaptor to 1/4 inch pipe thread for the gas regulator. If these items can be found in your laboratory, the kit need not be purchased.

Syringe Kit *(OIC Part #177-634)*

This kit includes two 5 ml syringes with 2-way valves for sample introduction. One of these syringes is included in the Startup Kit with the instrument, but stocking spares of all glass items is recommended.

GC Interface Kits

Included with the 4460A is a heated transfer line which carries desorbed volatiles to the GC. This transfer line must be connected to the GC column by means of plumbing interface. The 4460A is designed to operate with a variety of GC models, so GC Interface Kits are available for the connection of the transfer line to any of the popular gas chromatographs. Two types of GC Interface Kits are offered:

Carrier Interface Kit

This kit is comprised of hardware necessary to interrupt the GC carrier gas flow immediately downstream of the flow controller (or its hydrocarbon scrubber if present), re-route this gas to the 4460A carrier gas inlet, and re-connect the leftover GC injector carrier line to the 4460A transfer line. The carrier gas line to the GC injection port (which as the line disconnected from the flow controller) is wrapped

around the GC injector heater block if necessary, before connection to the transfer line. Thus, compounds desorbed from the 4460A move through the transfer line into the GC injection port, sweep past the injection port septum along with the carrier gas, and partition onto the GC column. The GC injection port can still be used for direct solvent injection of standards or extractions. The Carrier Interface Kit is suitable for use with 1/8 inch, 1/4 inch, or 5 mm OD packed columns.

Low-Dead-Volume Interface Kit

This kit includes the hardware necessary to interrupt the GC carrier gas flow immediately downstream of the flow controller and re-route the carrier gas to the 4460A like the Carrier Interface kit. Neither the GC carrier line to the injector nor the injector is used for gas flow, however. Rather, an extremely low-dead-volume injector kit is inserted through the GC injector and uses the existing injector only for mechanical support. The transfer line is connected to the inlet of this low-dead-volume injector and the column is connected to its outlet tube which protrudes into the GC column oven. The new injector also allows direct injection of standards or extractions, but only using a 2 inch long, 26 gauge syringe needle. A low-dead-volume union and ferrules for connection of standard and wide-bore capillary columns are included. The Low-Dead-Volume Interface Kit was designed for use with wide-bore fused silica columns, but is also suitable for use with 1/8 inch OD metal packed columns. In this case, a 1/16 x 1/8 inch union (not provided) must be used to connect the column to the protruding injector tube.

Chapter 2
Description of Components

In **Chapter 1** some basic concepts of 4460A operation, as well as its features and specifications are outlined. Also described are some additional hardware items available from O.I. Corporation, which may be either necessary for interfacing the concentrator to a gas chromatograph, or useful in its installation and operation.

This chapter deals with what we have named the various components of the 4460A and what the function of each is. Each significant component is pointed out and named on a photograph of one of the various views of the instrument. The function of each named component is also described, along with notes and cautions. The names are printed in **boldface type** in this chapter and are useful when you are trying to identify a part for ordering replacements.

Front Panel

A vessel for 5 ml samples is standard and one for 25 ml samples is optional. Optional 5 ml and 25 ml needle sparging vessels are also available.

Purge Vessel

Purge gas passes through this glass tube and is dispersed by the frit into the injected water sample. These purge gas bubbles act to strip the sample of volatile components and carry them from this vessel to the **trap**.

Sample Needle

This needle delivers a new sample to the bottom of the **purge vessel** and also acts as a drain line for the spent sample.

Sample Valve

This manual valve is used to direct sample flow from a syringe into the **sample needle**, or from the **sample needle** to the **drain line**. The arrow on the valve handle points toward the path of flow. When the arrow is pointed to neither the **syringe port** nor the **drain line**, the **sample needle** is isolated from any flow path.

Tee Fitting

This fitting contains Teflon ferrules at the top and bottom, to seal against the **sample needle** and the **purge vessel**, respectively. The **tee** side arm is the path for purge gas flow to the trap. This **tee** also physically supports the **purge vessel**.

Syringe Port

This Luer fitting allows connection of a syringe for delivery of water samples to the **purge vessel**.

Drain Line

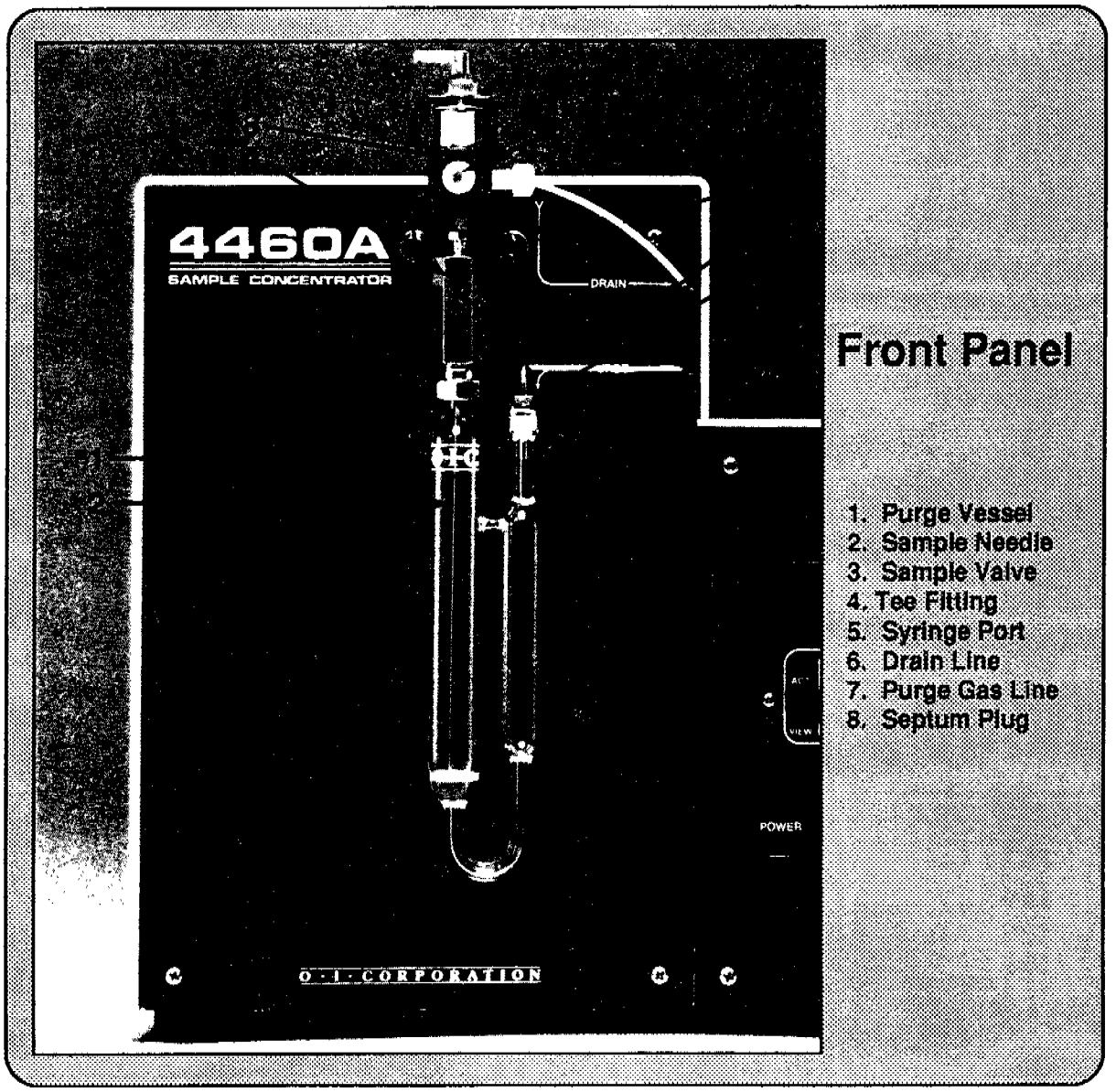
This line connects the **sample valve** to the **drain valve** for sample draining after purge.

Purge Gas Line

This line brings purge gas from the **purge valve** to the **purge vessel.**

Septum Plug

This Teflon-faced silicone rubber plug forms the seal between the **purge gas line** and the **purge vessel.** It should always be installed with the Teflon-face down into the **purge vessel** side arm.



Front Panel

1. Purge Vessel
2. Sample Needle
3. Sample Valve
4. Tee Fitting
5. Syringe Port
6. Drain Line
7. Purge Gas Line
8. Septum Plug

Keys and Displays

Power Switch

Power is turned on by pressing in the top of this key. Power status is indicated by a neon lamp in the switch.

Time Display

Time elapsed since beginning of each state of operation is displayed here. Time settings for each state can also be displayed. Time is displayed in MM:SS format.

Temperature Display

Actual and setpoint temperatures of various components are displayed here in degrees Celsius. Various sample counters can also be displayed here.

2nd FUNCT Switch

When this push button switch is pressed so that its red LED is lit, the second functions of certain front panel keys are enabled. Keys having second functions are marked with yellow labels describing that function of the key. The switch toggles between normal (LED off) and **2nd FUNCT** (LED on) status when alternately pressed.

AUTO - ON/OFF Key

This key toggles the system between automatic sequencing and manual sequencing through the states of operation. Its LED is lit when auto-sequencing is ON.

2nd FUNCT: When the **2nd FUNCT** LED is on, this key commands the system to jump directly to the **DRAIN** state. Once in **DRAIN**, you can start the timer to cycle the system through a timed **DRAIN** followed by a timed **BAKE**, then **STANDBY**. Times of **DRAIN** and **BAKE** are set as described in **TIMES Keys.**

RUN/STOP - STEP Key

When the **AUTO - ON/OFF** key is ON, this key toggles the timer on and off for the purpose of pausing during an auto-sequence. When the **AUTO - ON/OFF** key is OFF, this key is used to manually step the system through its various states.

2nd FUNCT: When the **2nd FUNCT** LED is on, this key commands the system to jump directly to **STANDBY**, and clears the timer and sample counters.

INC/DEC Switches

The leftmost of these two switches is used with the **TIMES SET** keys to change the default time settings. The rightmost switch is used with the **TEMPS SET** and **AUTO SPL SET** keys to change those default settings. Pressing the switch toward **INC** or **DEC** increases or decreases the value on the display by one unit. Holding the switch in that position causes rapid changes in the displayed value.

These time settings in memory can be increased or decreased by pressing the correspond- ing INC/ DEC switch up or down while viewing the TIME display.

New time settings do not take effect until another TIMES, TEMPS, or 2nd FUNCT key is pressed.

TIMES Keys

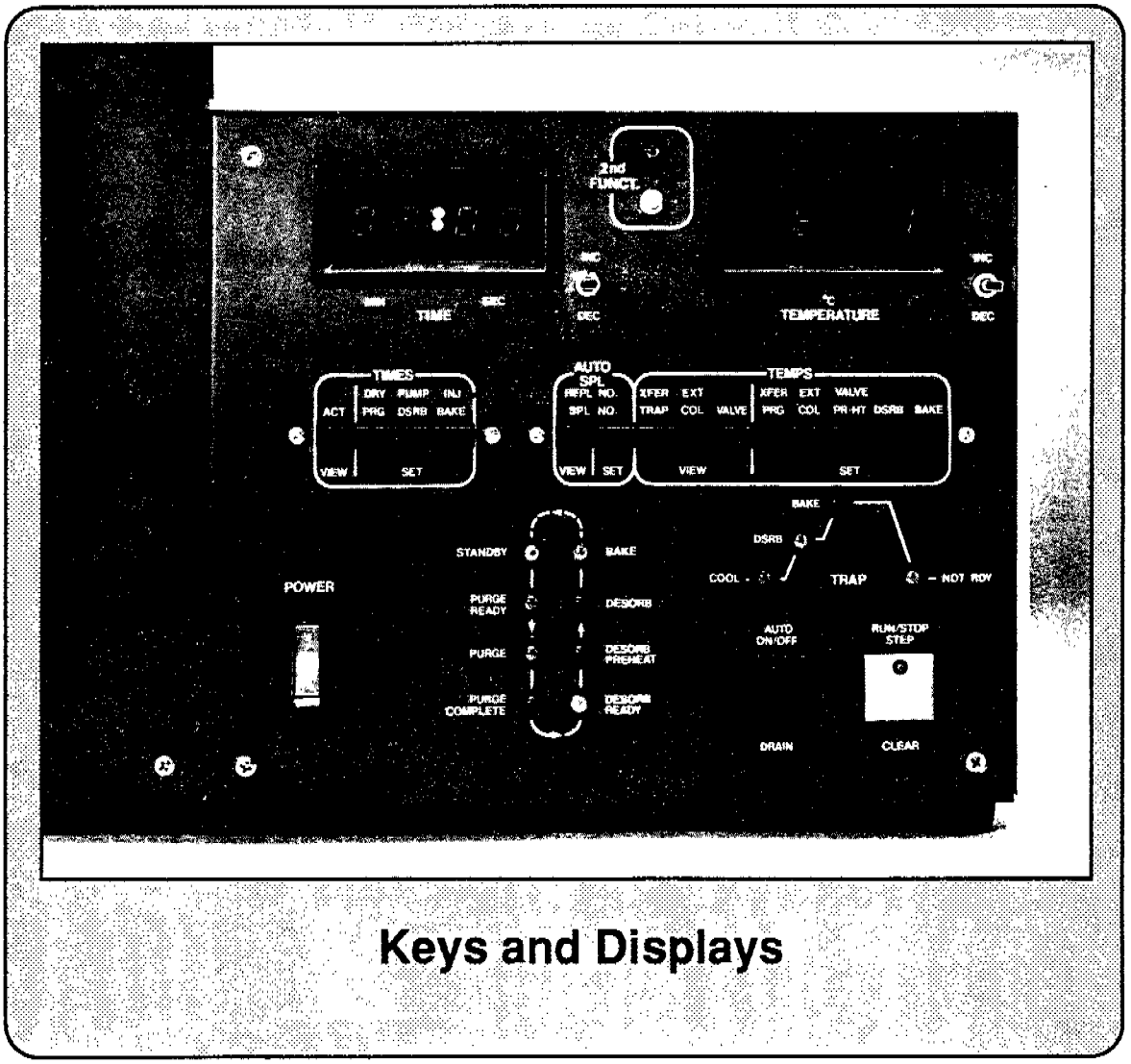
PRG/DRY SET

Pressing this key causes the **TIME** display to show the value in memory (MM:SS) for the time the system remains in **PURGE** during an auto-sequence.

2nd FUNCT: When the **2nd FUNCT** LED is on, the function of this key is changed to **DRY** and similarly affects **DRY PURGE**.

DSRB/PUMP SET

Pressing this key causes the **TIME** display to show the value in memory (MM:SS) for the time the system remains in **DESORB** during an auto-sequence.



Keys and Displays

2nd FUNCT: When the **2nd FUNCT** LED is on, the function of this key is changed to similarly affect the pump time for the autosampler during **LOOP FILL**. The **PUMP** setting affects the volume of sample which passes through the **sample loop** when the autosampler or other unattended analysis method is used.

The time the pump runs to pump rinse water during the autosampler **LOOP RINSE** is set at 20 seconds longer than the displayed **PUMP** time to achieve a more extended rinse of the loop during **LOOP RINSE 1** and **LOOP RINSE 2.**

BAKE/INJ SET

Pressing this key causes the **TIME** display to show the value in memory (MM:SS) for the time the system remains in **BACKFLUSH BAKE** during an auto-sequence.

2nd FUNCT: When the **2nd FUNCT** LED is on, the function of this key is changed to similarly affect the sample injection time for the autosampler during **LOOP INJECT**. The **INJ** setting affects the time allowed for injection of the sample loop volume into the **purge vessel** when the autosampler or other loop injection method is used.

The minimum time allowed for sample drain during **DESORB** and the time allowed for autosampler **DRAIN RINSE** is set at 15 seconds longer than the displayed **INJ** time to assure complete draining of sample/rinse from the **purge vessel.**

ACT VIEW

Pressing this key causes the **TIME** display to show the actual time elapsed since the beginning of the current state during an auto-sequence.

TEMPS Keys

These temperature settings in memory can be increased or decreased by pressing the corresponding INC/DEC switch up or down while viewing the TEMPERATURE display.

New temperature settings do not take effect until another TIMES, TEMPS, or 2nd FUNCT key is pressed.

PRG/XFER SET

Pressing this key causes the **TEMPERATURE** display to show the value in memory (°C) for the temperature setpoint of the **trap** during **PURGE.**

2nd FUNCT: When the **2nd FUNCT** LED is on, the function of this key is changed to similarly control the temperature of the **transfer line (XFER).**

COL/EXT SET

Pressing this key causes the **TEMPERATURE** display to show the value in memory (°C) for the temperature setpoint of the GC column. The automatic sequence will not advance past **PURGE COMPLETE** until the GC column temperature (as monitored by a thermocouple in the GC oven) decreases to this setpoint. This setpoint insures against premature desorption of a trapped sample onto the GC column. (A GC-ready signal from the GC to the 4460A can also be similarly used for this purpose. Refer to **Rear Panel Terminal Strip**).

CAUTION
The GC thermocouple must be properly connected to the 4460A and placed in the GC oven for this function to operate properly.

2nd FUNCT: When the **2nd FUNCT** LED is on, the function of this key is changed to control the temperature of an external heated zone. Pressing the key in this manner causes the **TEMPERATURE** display to show the value in memory (°C) for the temperature setpoint of an external heated device. The external heated device may use power from the 4460A or from an external power source. If power from the 4460A is used, the temperature of the external heated device can be regulated at a setpoint as well as monitored by the 4460A.

PR-HT/VALVE SET

Pressing this key causes the **TEMPERATURE** display to show the value in memory (°C) for the temperature setpoint of **DESORB PREHEAT**. The automatic sequence will not advance past **DESORB PREHEAT** to desorb the trapped sample until the trap temperature increases to this setpoint.

2nd FUNCT: When the **2nd FUNCT** LED is on, the function of this key is changed to control the temperature of the **valve oven** internal to the 4460A. Pressing the key in this manner causes the **TEMPERATURE** display to show the value in memory (° C) for the temperature setpoint of the **valve oven.**

DSRB SET

Pressing this key causes the **TEMPERATURE** display to show the value in memory (°C) for the temperature setpoint of the trap during **DESORB** .

When a jumper is plugged in place of a thermocouple for any of these devices, the temperature will read ambient.

BAKE SET

Pressing this key causes the **TEMPERATURE** display to show the value in memory (°C) for the temperature setpoint of the trap during **BACKFLUSH BAKE** .

TRAP/XFER VIEW

Pressing this key causes the **TEMPERATURE** display to show the actual temperature (°C) of the trap.

2nd FUNCT: When the **2nd FUNCT** LED is on, the function of this key is changed to **XFER** and displays the actual temperature (°C) of the **transfer line.**

COL/EXT VIEW

Pressing this key causes the **TEMPERATURE** display to show the actual temperature (°C) of the GC column.

2nd FUNCT: When the **2nd FUNCT** LED is on, the function of this key is changed to **EXT** and displays the actual temperature (°C) of the external heated zone.

VALVE VIEW

Pressing this key causes the **TEMPERATURE** display to show the actual temp (°C) of the **valve oven.**

AUTOSPL Keys

SPL NO./REPL NO. SET

Pressing this key causes the **TEMPERATURE** display to show the value in memory for the setpoint number of samples to be analyzed during unattended analysis. This **SPL NO.** setpoint is generally used in conjunction with the autosampler.

2nd FUNCT: When the **2nd FUNCT** LED is on, the function of this key is changed to **REPL NO.** and similarly controls the number of replicates to be analyzed from each autosampler vial.

These setpoint values in memory can be increased or decreased by pressing the correspondng INC/ DEC switch up or down while viewing the TEMPERATURE display.

New counter settings do not take effect until another TIMES, TEMPS, or 2nd FUNCT key is pressed.

SPL NO./REPL NO. VIEW

Pressing this key causes the **TEMPERATURE** display to show the sample number of the sample presently being analyzed. This sample number will increase by one with each new sample until the **SPL NO. SET** value is reached. Upon completion of the last analysis, the **SPL NO. VIEW** will match the **SPL NO. SET** and the 4460A will hold in **STANDBY**.

2nd FUNCT: When the **2nd FUNCT** LED is on, the function of this key is changed to display the replicate number of the sample presently being analyzed. This replicate number will increase by one with each new replicate until the **REPL NO. SET** value is reached. Upon completion of the last replicate from an autosampler vial, the 4460A will cycle the entire system through its automatic rinses, increment the **SPL NO.** by one, and begin analysis of the set number of replicates from the next vial.

STATE STATUS LEDs

Primary States - The 4460A is in one of the primary states (**STANDBY, PURGE READY, PURGE, PURGE COMPLETE, DESORB READY, DESORB PRE-HEAT, DESORB W/DRAIN**, and **BACKFLUSH BAKE**) if this corresponding LED is on.

Dry Purge - The 4460A is in **DRY PURGE** if the **PURGE** LED is blinking.

Loop Rinses - The 4460A is in its autosampler **LOOP RINSE** sequence if both the **DESORB** and **BAKE** LEDs are blinking.

Drain - The 4460A is in **DRAIN** if the **BAKE** LED is blinking.

Desorb w/o Drain - The 4460A is in **DESORB W/O DRAIN** if the **DESORB** LED is blinking.

TRAP STATUS LEDs

The status of the trap temperature is indicated by these LEDs. When the LED corresponding to **COOL, DSRB** or **BAKE** is fully on, the trap temperature is being held at its setpoint. When an LED is blinking, the trap temperature is approaching the corresponding temperature setpoint.

Left Bay

Valve Oven

This oven houses the heated components of the 4460A, including the **trap valve** and the **trap bulkhead fittings**. It is described in detail later in this chapter under its own section. The oven cover is removed by turning the 1/4-turn latch to its vertical position and pulling outward.

Trap

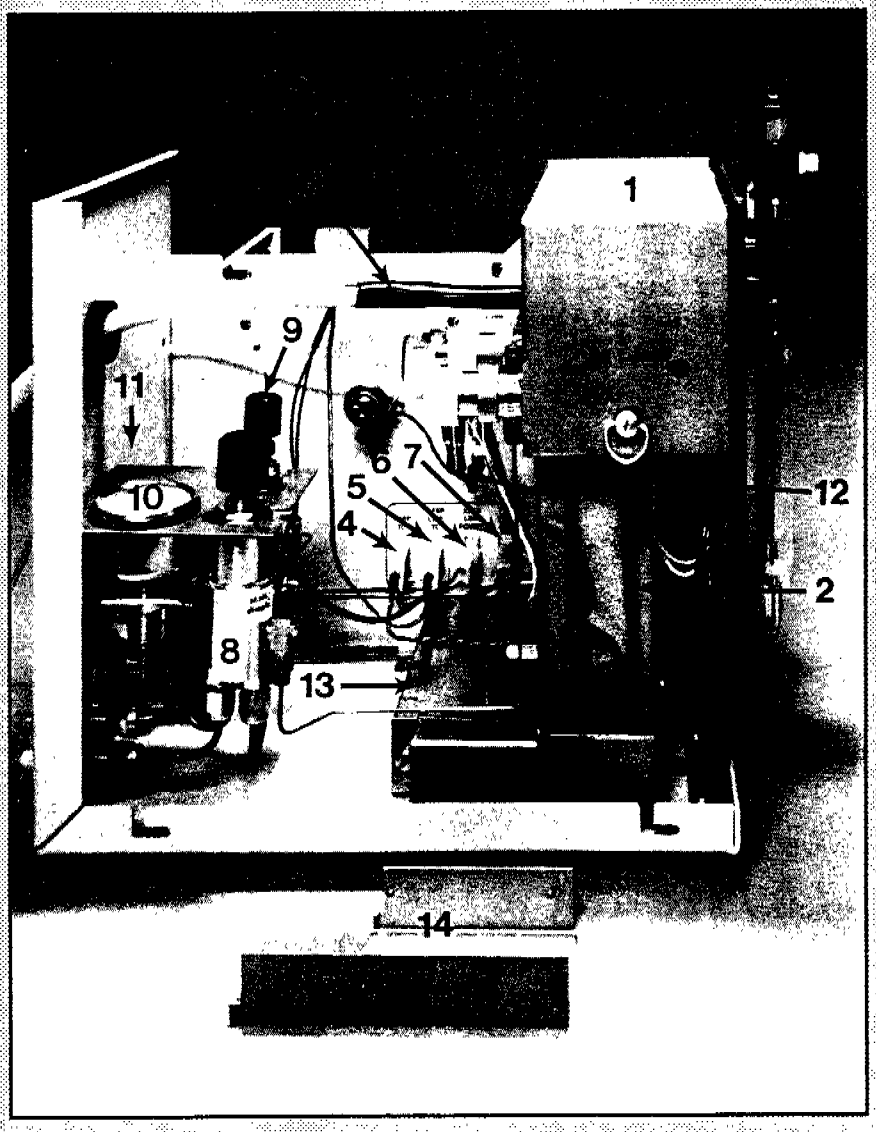
The **trap** is a stainless steel tube, 0.125 inches OD x 0.105 inches ID and 12 inches long (3.2 mm x 2.7 mm x 30.5 cm), packed with a series of adsorbents for trapping purgeable organic compounds. The tube has a Type K thermocouple welded to it for

temperature feedback and control. The metal tube is rapidly heated by induction of an electrical current through it, and is cooled by forced air pumped from beneath the unit.

Four different adsorbent packing arrangements are available. These are:

Trap #1 Tenax Only (OIC Part #169559)
Trap #2 OV-1/Tenax (OIC Part #169567)
Trap #3 OV-1/Tenax/Silica Gel (OIC Part #169517)
Trap #4 OV-1/Tenax/Silica Get/Charcoal (OIC Part #169525)

·"OV-1" is a methyl silicone packing of 3% OV-1 on 60/80 mesh Chromosorb-W or equivalent.



LEFT BAY

1. Valve Oven
2. Trap
3. Transfer Line
4. Trap TC Receptacle
5. Xfer Line TC Recptacle
6. Xfer Line Power Recptacle
7. Fan 1 Power Receptacle
8. System Pressure Regulator
9. Purge Gas Flow Controller
10. Purge Gas Pressure Gauge
11. Trap Valve Actuator Solenoid
12. Trap Valve Actuator
13. Fan
14. Fan Duct Cover

- "Tenax" is 2,6-Diphenylene oxide polymer, 60/80 mesh, chromatographic grade or equivalent.
- "Silica Gel" is 35/60 mesh silica gel, Davison, grade-15 or equivalent.
- "Charcoal" is coconut charcoal, 6/10 mesh sieved to 26 mesh, Barnaby Chaney, CA-580-26 lot #M-2649 or equivalent.

All traps are packed as specified by current EPA Methodology.[1] Each trap has the corresponding trap number stamped on the nut on the INLET side and a "0" stamped on the OUTLET side. A trap with no number stamped on it contains no packing when shipped from the factory.

[1] *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule, Environmental Protection Agency, 40 CFR Part 136, Federal Register, Vol. 49, No. 209, October 26, 1984.*

Transfer Line

This is a fused-silica-lined stainless steel tube, .062 inches OD x 0.020 inches ID and 48 inches long (1.6 mm x 0.5 mm x 122 cm); surrounded by a Teflon jacket and heating coils. Power and thermocouple plugs are connected to receptacles in the 4460A. The temperature of this line can be set from ambient to 150°C. The **transfer line** tube can be easily extracted from its Teflon jacket and replaced by other tubing if desired.

CAUTION:
Thermocouple receptacles must have either a thermocouple or jumper installed for the temperature readout to operate properly.

Trap TC Receptacle

This thermocouple receptacle receives the **trap** thermocouple for trap temperature control and regulation. It is designed for easy disconnection during trap replacement.

Xfer Line TC Receptacle

This thermocouple receptacle receives the **transfer line** thermocouple for transfer line control and regulation.

Xfer Line Power Receptacle

This power receptacle receives the AC power plug of the **transfer line.**

Fan 1 Power Receptacle

This receptacle receives the AC power plug of the **trap fan.**

System Pressure Regulator

This regulator is normally set to 20 psi (138 kPa) output and regulates the working gas pressure used by the 4460A system including the autosampler.

Purge Gas Flow Controller

This flow controller acts to regulate the flow of purge gas even if the restriction caused by the **trap** changes with time.

Purge Gas Pressure Gauge

This pressure gauge displays the pressure required to sustain the purge gas flow rate through the **trap** as regulated by the **purge gas flow controller.**

Trap Valve Actuator Solenoid

This 4-way solenoid valve distributes gas pressure to the **trap valve actuator** upon electrical command for **trap valve** rotation. The red LED indicates when the solenoid is energized (when the **trap valve** is rotated to the activated position). The **trap valve** may also be rotated by pressing the manual actuation button on top of this solenoid.

Trap Valve Actuator

This pneumatic actuator rotates the **trap valve** when gas pressure is applied to one of two actuator input ports and relieved from the other. This pressure is directed by the **trap valve actuator** solenoid. The actuator rotates the **trap valve** 90° clockwise to the activated position.

Fan

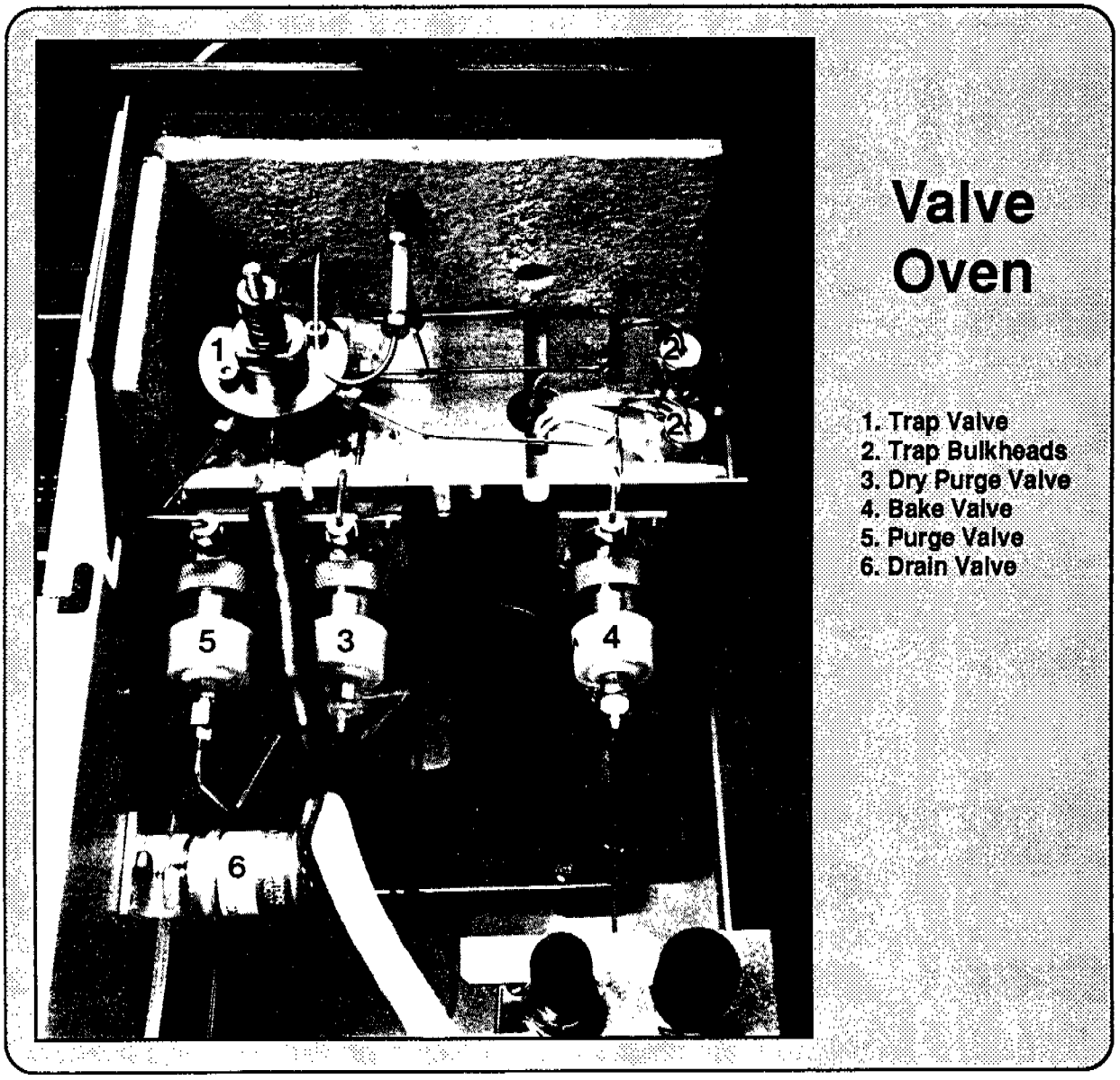
This fan pulls air from beneath the 4460A to cool the trap when the trap is to be maintained at the purge temperature. The fan is turned off when the trap is to be maintained at the desorb or bake temperatures. Thus, the fan cycles on and off during an analysis sequence. The air circulation also cools the 4460A.

Fan Duct Cover

This cover helps duct the cooling air across the trap. It is removed by simply pulling outward to expose the trap for replacement.

Valve Oven

The components inside the **valve oven** can be maintained at temperatures ranging from ambient to 200° C. All of the fluid-flow lines between the **purge vessel, trap,** and **transfer line** are contained in this oven, and the volume seen by the compounds of interest has been minimized, so that peak-tailing and carry-over problems have been essentially eliminated. These features also help make the 4460A more compatible with wide-bore capillary columns.



Trap Valve

This 6-port valve allows transfer of trapped compounds from the **trap** to the **transfer line.** It rotates 90° from **trapping** position (fully CCW) to **desorbing** position (fully CW) by means of a pneumatic actuator.

Trap Bulkheads

These bulkhead fittings provide mechanical and electrical connection for the **trap.**

Dry Purge Valve

When activated, this 3-way solenoid valve routes the purge gas directly through the **trap,** bypassing the **purge vessel.**

Bake Valve

This 3-way solenoid valve provides a flow of backflush gas to the **trap** during **BACKFLUSH BAKE.**

Purge Valve

When activated, this 3-way solenoid valve provides purge gas to the **purge vessel.**

Drain Valve

When activated, this 3-way solenoid valve opens flow from the **purge vessel** to allow the draining of the water sample or rinse solution.

Right Bay

Program E/PROM

This EPROM (Erasable-Programmable-Read-Only-Memory) integrated circuit contains the entire system operating software. It contains 8K bytes of machine language code which is needed and executed by the system microprocessor. Field software upgrade is possible by simply un-plugging this device and installing a new version **Program EPROM** when power is off.

Microprocessor

This is the CPU (Control Processor Unit) for all instrument functions. It is an 8085 type device, and operates at a rate of 2.6 million clock cycles per second.

Temperature Control Adjustment Pot

This potentiometer adjusts the accuracy of the ADC (analog to digital convertor) chip used for conversion of the multiplexed analog thermocouple signals.

DIP Switches

These seven **DIP switches** have the following functions:

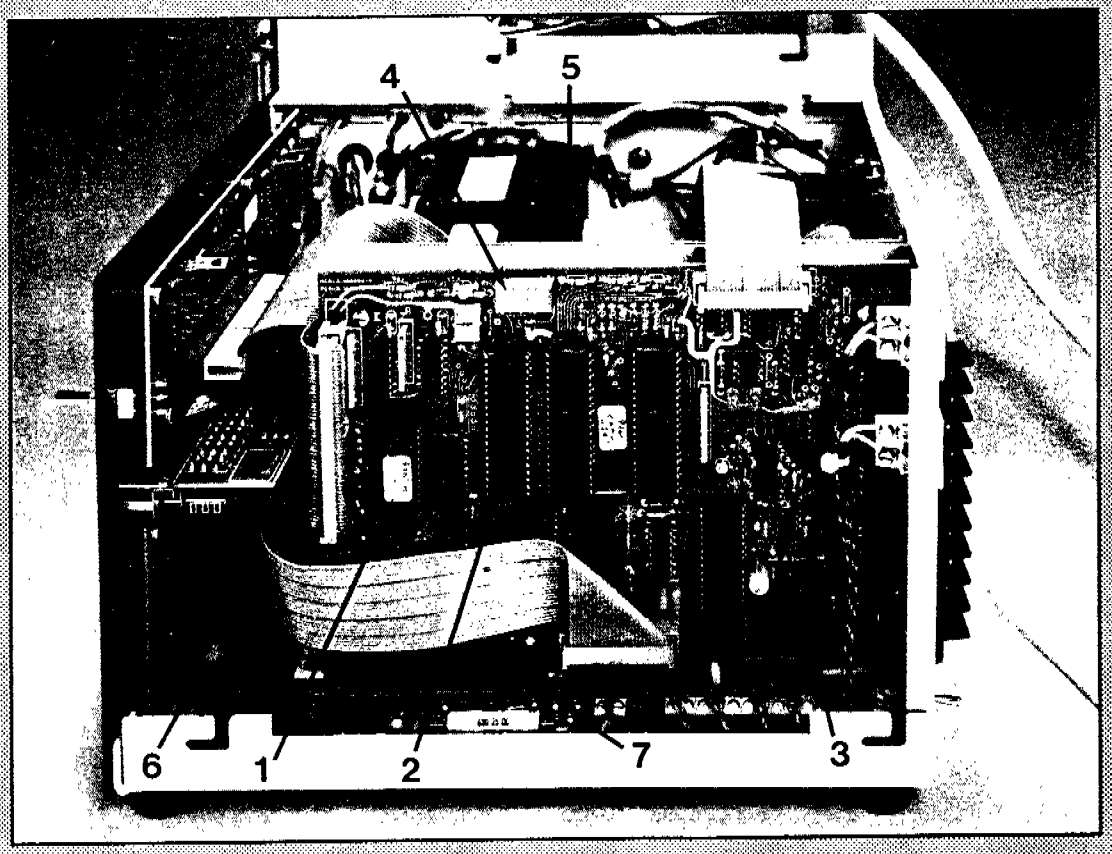
DIP Switches 2, 3, and 4 are used in combination to select one of eight different sets of time and temperature setpoints. Currently, there are four different sets of times and temperatures found in US EPA purge-and-trap methodologies. Each of these unique sets has been assigned a Method Designator. The following tables define these Method Designators, each of which can be selected using the specified combination of DIP switch settings.

Switch #	Function
1 ·	Autosampler is enabled when this switch is ON
2 ·	Method selection (see tables below)
3 ·	Method selection (see tables below)
4 ·	Method selection (see tables below)
5 ·	Relay closure occurs at beginning of **DESORB** when this switch is OFF, at end of **DESORB** when ON. This switch is normally set to OFF.
6 ·	When AUTO is on with timer running: timer stops at **PURGE READY** when this switch is ON; timer cycles automatically from **PURGE READY** to **PURGE** when this switch is OFF. This switch is normally set to ON when syringe-injecting samples.
7 ·	When AUTO is on with timer running: timer stops at **DESORB READY** when this switch is ON; timer cycles automatically from **PURGE COMPLETE** to **DESORB PREHEAT** when this switch is OFF. When this switch is set to ON, it is normally used in conjunction with feedback from the GC. When it is set to OFF, feedback from the GC in the form of sensing the oven temperature with a thermocouple is needed.

Method Designator	EPA Methods Included	Dip Switch Settings 2	3	4
601	502.1, 524.1, 601, 624	Off	Off	Off
503	503.1	Off	Off	On
602	602	Off	On	Off
603	603	Off	On	On

Method Designator	------State Times (min)------ PURGE · DRY PURGE · DESORB · BAKE			Trap Temps (°C) Prg	Dsrb	Bake
601	11.0	0.0	4.0	7.0	25	180	180
503	12.0	4.0	4.0	7.0	25	180	180
602	12.0	6.0	4.0	7.0	25	180	180
603	15.0	0.0	1.5	7.0	85	180	210

To select one of these methods, simply set DIP switches 2, 3, and 4 to the desired settings, power down, and power back up. The Method Designator will then be displayed in the **TEMPERATURE** display for 10 seconds.



RIGHT BAY

1. Program E/PROM
2. Microprocessor
3. Temperature Control Adjustment Pot
4. Dip Switches
5. Voltage Select Plug
6. DC Power Status LEDs
7. DC Control Status LEDs

WARNING:
Do not connect unit to 230 VAC supply unless the proper voltage has been selected.

Voltage Select Plug (not in view)

This plug selects the line voltage from which the unit will be operated. It is located on the motherboard at the bottom of the right bay. Operating line voltage (115 vs 230 VAC) is selected by connecting the jumper to the appropriate connector as marked on the motherboard. Line frequency selection is automatic and requires no equipment user selection.

DC Power Status LEDs (not all in view)

These four LEDs on the motherboard show the operational status of the four regulated power supplies. These are illuminated if proper DC voltages are present in the system (+5 V digital, +5 V analog, +12 V control).

DC Control Status LEDS

These eight LEDs indicate when +12 V DC power is being applied to each of the various +12 volt operated devices in the left bay or autosampler (valves, solenoids, etc.). They are provided for convenience during functional testing the instrument. Their device designation is as follows:

Position	LED #	Function
Rearmost	PA0	Purge Valve
	PA1	Drain Valve
	PA2	Injection Gas Valve (LSM)
	PA3	Loop Valve (LSM)
	PA4	Trap Valve
	PA5	Dry Purge Valve
	PA6	Bake Valve
Frontmost	PA7	Sample Pump and Fill Gas Valve (LSM)

Rear Panel

Purge Gas Inlet

This fitting is a 1/8 inch Swagelok unión for accepting the purge gas inlet line. The nut and ferrule set for this union are included.

Actuator Gas Inlet

This barbed fitting accepts a 1/8 inch OD x 1/16 inch ID (3.2 x 1.6 mm) plastic or Teflon tube for supplying the actuator gas.

Carrier Gas Inlet

This fitting is a 1/16 inch (1.6 mm) OD tube Valco bulkhead union for accepting the carrier gas line from the GC flow controller. The nut and ferrule for this union are included.

Hydrocarbon Scrubber

This scrubber consists of a stainless steel tube filled with 13X molecular sieve for trapping organic contaminants in the purge gas stream. It has been baked at 300°C under inert gas flow for at least two hours at the factory.

Autosampler Gas Outlet

This fitting is a 1/16 inch (1.6 mm) OD tube Valco bulkhead union for providing gas to the **Loop Sampling Module** (LSM).

Transfer Line Feedthrough

This hole is for feedthrough of the heated **transfer line** from the **valve oven** to the GC injection port.

Drain Line Feedthrough

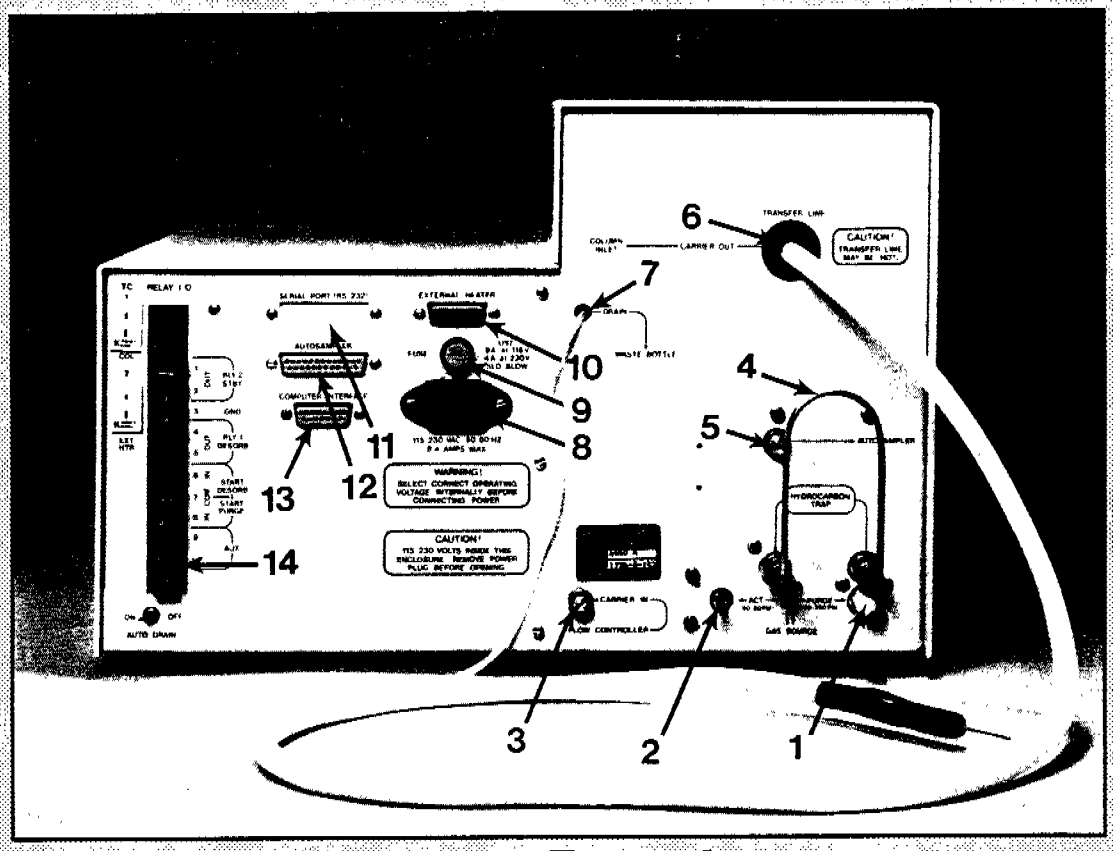
This hole is for feedthrough of the sample drain line from the **drain valve** to a waste receptacle.

Main AC Power Receptacle

This receptacle receives 115 VAC or 230 VAC power by means of a modular power cord, for operation of the 4460A.

Main AC Power Fuse

This fuse provides overall protection against damage due to electrical malfunction. Use an 8 amp slo-blow for 115 VAC operation and 4 amp slo-blow for 230 VAC operation.



Rear Panel

1. Purge Gas Inlet
2. Actuator Gas Inlet
3. Carrier Gas Inlet
4. Hydrocarbon Scrubber
5. Autosampler Gas Outlet
6. Transfer Line Feedthrough
7. Drain Line Feedthrough
8. Main AC Power Receptacle
9. Main AC Power Fuse
10. External Heated Zone Power Receptacle
11. Serial Port
12. Autosampler Cable Receptacle
13. Computer Interface Receptacle
14. Terminal Strip-Relay I/O

External Heated Zone Power Receptacle

This receptacle provides AC power to an externally-located heating device by means of a mating connector and cable. This connector and cable are not provided as standard with the 4460A, but are available from OIC.

Serial Port (RS-232) Receptacle (Optional)

When this option is ordered from the factory this receptacle is provided for connection to the RS-232 serial port communication. It mates with a standard female DB-25 serial connector when this option is ordered from the factory.

Autosampler Cable Receptacle

This receptacle provides both TTL level signals and 12 VDC control signals for operation of the autosampler system (LSM and ASM) by means of the **4460A/LSM Interface Cable.**

Computer Interface Receptacle

This receptacle provides four opto-isolated inputs and outputs for external direct control of the instrument allowing for future expansion of the system.

Terminal Strip - Relay I/O

This terminal strip provides electrical I/O connections for interfacing the 4460A with the GC, integrator, and/or data system. The function of each of the terminals is as follows:

Terminals 1 and 2: STANDBY Relay Output (Relay 2)

A two-second closure occurs across terminals 1 and 2 upon power up and return of system to **STANDBY**. This output is typically used to communicate the completion of a 4460A cycle. The relay closure is rated at 120 V and 5 amps maximum.

Terminal 3: Ground

Terminals 4 and 5: DESORB Relay Output (Relay 1)

A two-second closure occurs across terminals 4 and 5 at the beginning or end of **DESORB**, depending on the setting of **DIP switch #5**. The normal setting of this DIP switch is OFF, to allow a closure at the beginning of **DESORB** for start of an integrator and/or GC temperature program. The relay closure is rated at 120 V and 5 amps maximum.

Terminals 6 and 7: Start DESORB Input

A two-second contact closure across terminals 6 and 7 will cause the system to advance from **DESORB READY** to **DESORB PRE-HEAT** when **DIP switch #7** is ON. This "hold" at **DESORB READY**, using DIP switch #7, is typically used to insure that the GC has completed its temperature-programmed column cool-down (GC-ready) before the next sample is desorbed from the trap onto the GC column. (The GC column thermocouple may also be used for this purpose using **PURGE COMPLETE.**

When DIP switch #6 or #7 is OFF, the system advances automatically past the affected state regardless of any contact closure or shorting of these terminals.

If these two terminals are shorted together, the system will advance from **DESORB READY** to **DESORB PRE-HEAT** automatically even when DIP switch #7 is ON.

Terminals 7 and 8: Start PURGE Input

A two-second contact closure across terminals 7 and 8 will cause the system to advance from **PURGE READY** to **PURGE** when DIP switch #6 on ON. This "hold" at **PURGE READY**, using DIP switch #6, is typically used so that the next sample can be manually injected before the system advances automatically into **PURGE**.

If these two terminals are shorted together, the system will advance from **PURGE READY** to **PURGE** automatically even when DIP switch #6 is ON.

Column Thermocouple Receptacle - TC1

This receptacle houses the connection for a Type K thermocouple which can be inserted into the GC oven in order to monitor the column temperature.

External Heater Thermocouple Receptacle - TC2

This receptacle houses the connection for a Type K thermocouple which can be used to provide temperature control for an external heated device. It is used in conjunction with the **External Heated Zone Power Receptacle.**

Auto Drain Switch

When ON, this switch allows the **drain valve** to open during **DESORB** and **DRAIN** for automatic draining of sample from the **purge vessel.** When OFF, the switch allows the sample to remain in the **purge vessel** during **DESORB** so that it can be re-purged (typically for purge efficiency experiments). The corresponding **DESORB** states are called **DESORB W/DRAIN** and **DESORB W/O DRAIN.**

Loop Sampling Module (LSM) - Front Panel

WARNING:
The needle will lower regardless of its lateral position so the user should be ready for it to pierce any vial septum or finger located underneath it.

RAISE Key

Pressing this key causes the **actuator arm** and **septum piercing needle assemblies** of the **Autosampling Module** to raise. Once the actuator arm has raised, pressing this key causes the needle to swing to a position over the sample vial. It can be used to manually override the automatic sampling sequence.

LOWER Key

Pressing this key causes the **actuator arm** and **septum piercing needle assemblies** at the **Autosampling Module** to lower. It can be used to manually override the automatic sampling sequence.

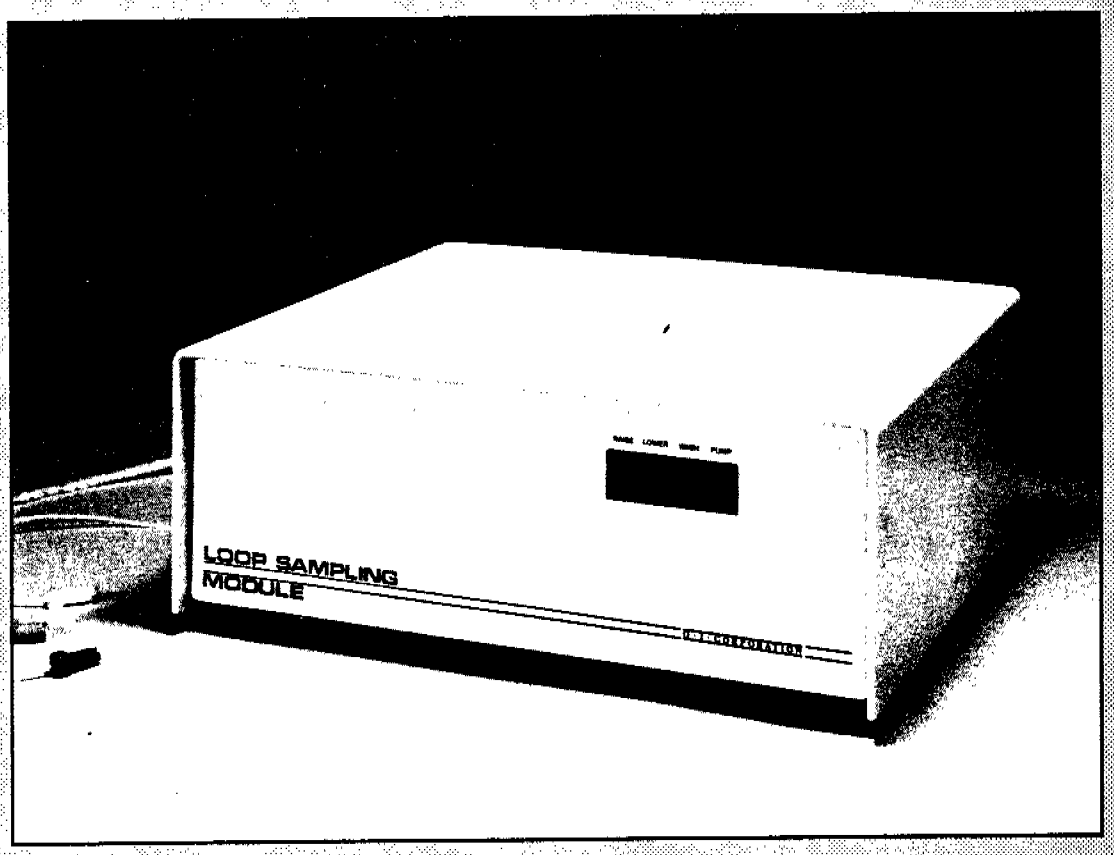
Pressing the RAISE key will swing the needle back to position over the sample vial when the actuator arm is raised.

WASH Key

Pressing this key causes the **septum piercing needle assembly** to swing laterally to a position over the **wash vessel** if the **actuator arm assembly** is in its raised position. It can be used to manually override the automated sampling sequence.

PUMP Key

Pressing this key causes the **sample pump** inside the LSM to run. When the **septum piercing needle** is immersed in water, the key allows manually activated pumping of the water through the **sample loop.** The **sample pump** will continue to run until the key is pressed again.



Loop Sampling Module - Front Panel

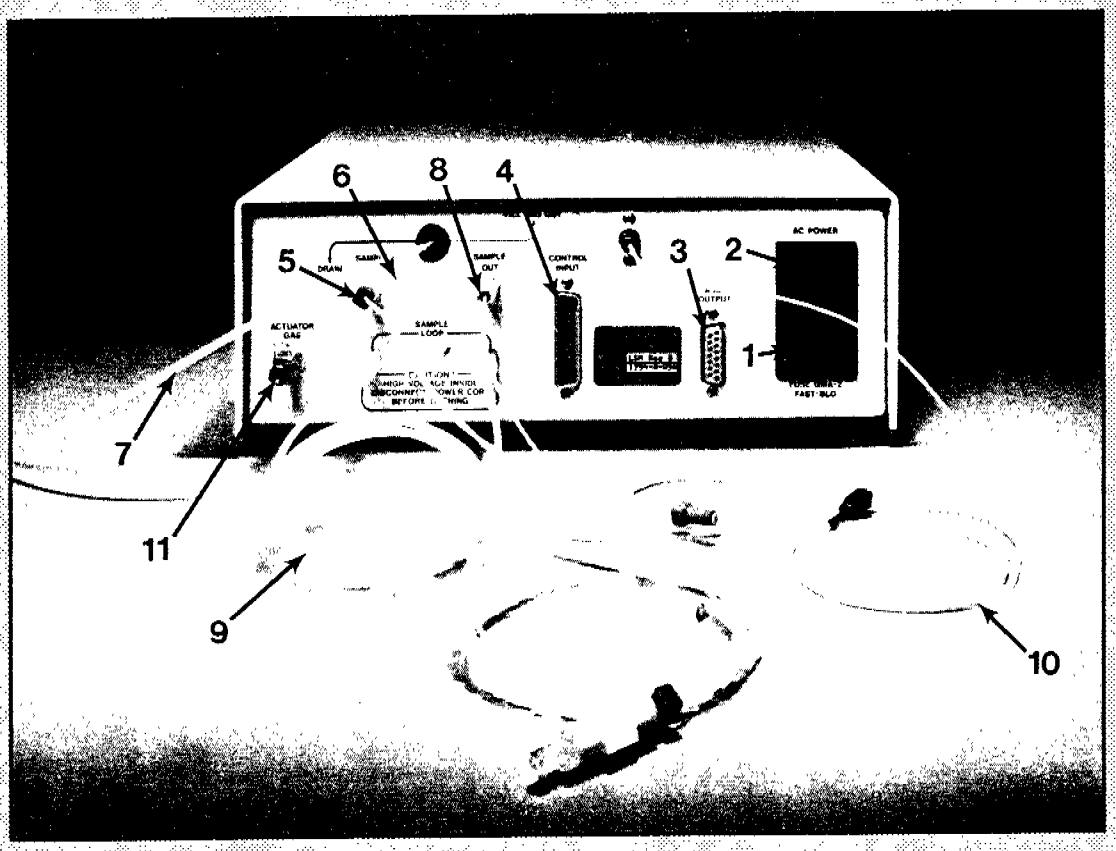
Loop Sampling Module (LSM) - Rear Panel

LSM Power Receptacle

This receptacle receives 115 VAC or 230 VAC power by means of a modular power card for operation of the **LSM** and **Autosampling Module** (ASM). It also houses one active and one spare 2A fuse.

Power Output Receptacle

This receptacle provides 115 VAC or 230 VAC power to the ASM by means of a modular power cord connected between the two modules.



Loop Sampling Module - Rear Panel

1. LSM Power Receptacle
2. Power Output Receptacle
3. ASM Output Receptacle
4. Contol Input Receptacle
5. LSM Sample Inlet Line
6. LSM Fill Gas Outlet
7. Drain Line
8. LSM Sample Outlet Line
9. Sample Loop
10. LSM Fill Gas Inlet Line
11. LSM Actuator Gas Line

ASM Output Receptacle

This receptacle connects the LSM to the ASM by means of a round **LSM/ASM interface cable** and provides TTL level signals for ASM operation.

Control Input Receptacle

This receptacle receives both TTL level signals and 12 VDC control signals for operation of the autosampler system (LSM and ASM) by means of the **4460A/LSM interface cable.**

LSM Sample Inlet Line (Blue Nut)

Through this line flows the liquid sample or rinse solution from **septum piercing needle** of the ASM into the **sample loop** of the LSM.

LSM Fill Gas Outlet Line (Clear Nut)

This line provides gas pressure to the sample vial using the **septum piercing needle assembly** outer needle, to force liquid from the vial up through the inner needle and out the LSM **sample inlet line.**

Drain Line

Through this line flows the excess sample from the **sample loop** to a waste vessel or drain.

LSM Sample Outlet Line (Red Nut)

Through this line flows the sample from the **sample loop** to the **4460A purge vessel.** The tee on its end connects the **LSM sample outlet line** to the 4460A **sample valve,** while still providing a drain for spent sample in the **purge vessel.** The line from the LSM is marked by red nuts on each end.

Sample Loop

This tubing has been calibrated to deliver 5.0 ml of sample to the 4460A **purge vessel** when used in conjunction with the **sample loop valve.** Loops of 25.0 ml calibrated volume are also available and volumes between 1 and 25 ml can be custom fabricated.

LSM Fill Gas Inlet Line (Blue Band)

This line provides gas pressure to the LSM from the 4460A for filling and injecting the **sample loop.** It is marked by a blue band near the nut. Its small ID provides a restriction to the 20 psi applied to it by the 4460A so that about 40 ml/min is produced at the LSM end.

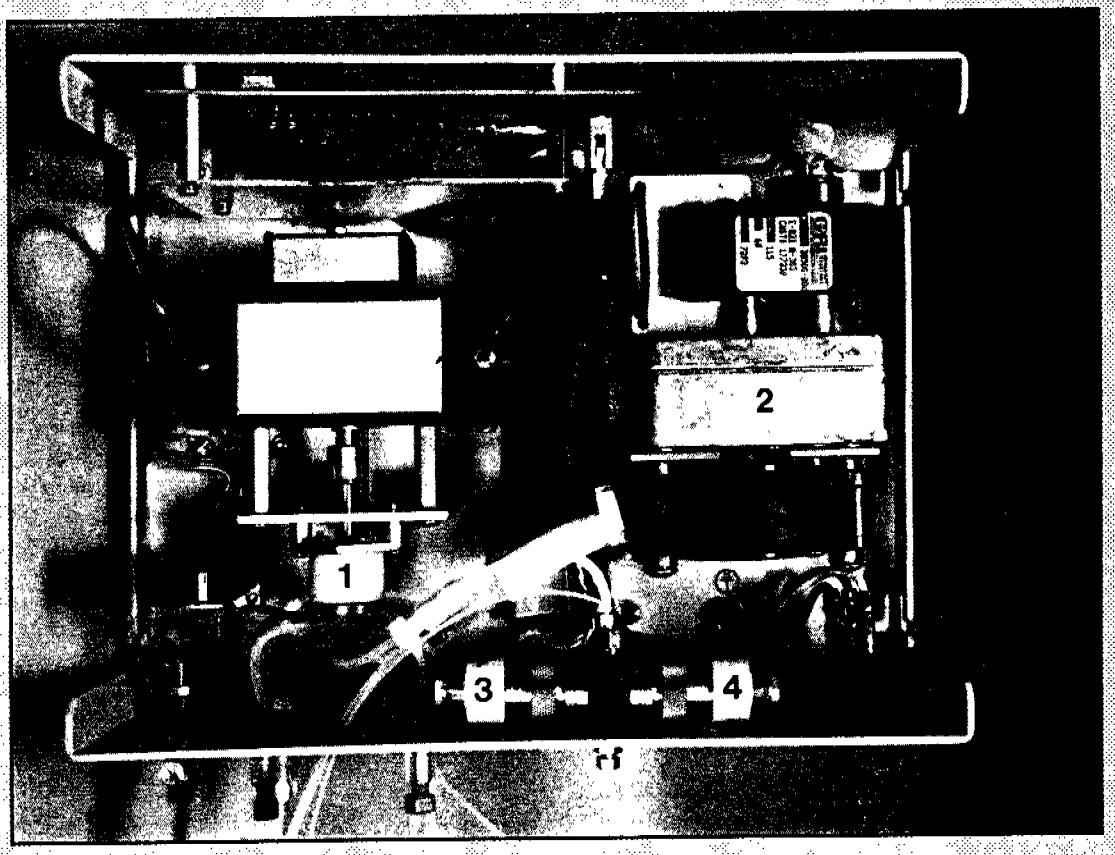
LSM Actuator Gas Line

Gas pressure for operating the **sample loop valve** inside the LSM is connected here. Pressure requirements are the same as for the 4460A actuator gas (40-80 psi) so these two inlets can be connected together.

Loop Sampling Module (LSM) - Interior

Sample Loop Valve

When deactivated, this valve allows flow from the **septum piercing needle** through the **sample loop**, through the **sample pump**, then to drain. When activated it redirects the contents of the **sample loop** into the 4460A **purge vessel.**



Loop Sampling Module - Interior

1. Sample Loop Valve
2. Sample Pump
3. Fill Gas Valve
4. Loop Inject Valve

Sample Pump

This pump serves three functions: (1) when gas pressure pushes liquid from the sample vial through the **sample loop,** the downstream pump works to regulate the rate of liquid flow by its peristaltic action. (2) When a replicate sample has been taken into the sample loop, the still pump acts as a downstream plug to help keep the sample under pressure until it can be injected into the **purge vessel.** (3) During washes between samples, the pump works to pull rinse solution from the **wash vessel** through the sample loop, through the pump, and out to drain.

Fill Gas Valve

When activated, this valve opens gas flow from the 4460A to provide pressure for filling the **sample loop.**

Loop Inject Valve

When activated, this valve opens gas flow from the 4460A to push sample through the **sample loop** and into the **purge vessel.**

Autosampling Module (ASM) - Front View

Rotary Tray

This tray is designed to hold **vial racks** of various sizes and will rotate the racks consecutively to bring successive sample vials to the sampling station underneath the **septum piercing needle.**

Vial Racks (Set of Nine)

These racks hold standard 22 mm OD (40 ml) VOA vials. There are three vial positions per rack and nine racks fit on the rotating tray (27 vials total). Racks for 18 mm OD, 16 mm OD, and other vials are also available.

Actuator Arm Assembly

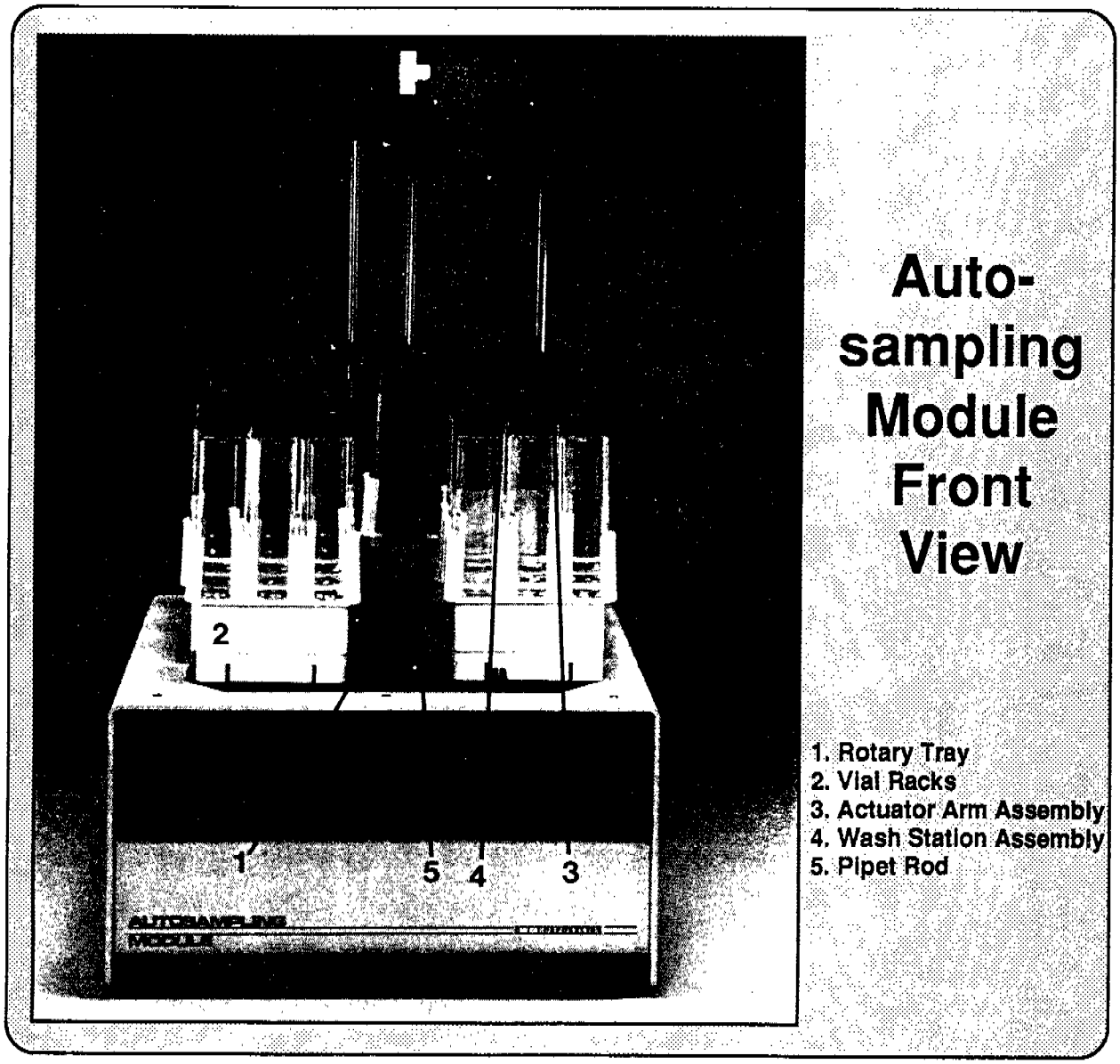
This mechanism moves this **septum piercing assembly** back and forth from the sample vial to the **wash vessel.**

Wash Station Assembly

This assembly is used to hold the **wash vessel** and to position the **septum piercing needle** properly over the sample vials.

Pipet Rod

This rod raises and lowers the **actuator arm assembly.**



Auto-sampling Module Front View

1. Rotary Tray
2. Vial Racks
3. Actuator Arm Assembly
4. Wash Station Assembly
5. Pipet Rod

Autosampling Module (ASM) Rear View

ASM Power Receptacle

This receptacle receives 115 VAC or 230 VAC power from the LSM by means of a short modular ASM power cord.

Manual Advance Button

This button is used to manually advance the rotary tray in order to move a selected sample vial into position.

Reset Button

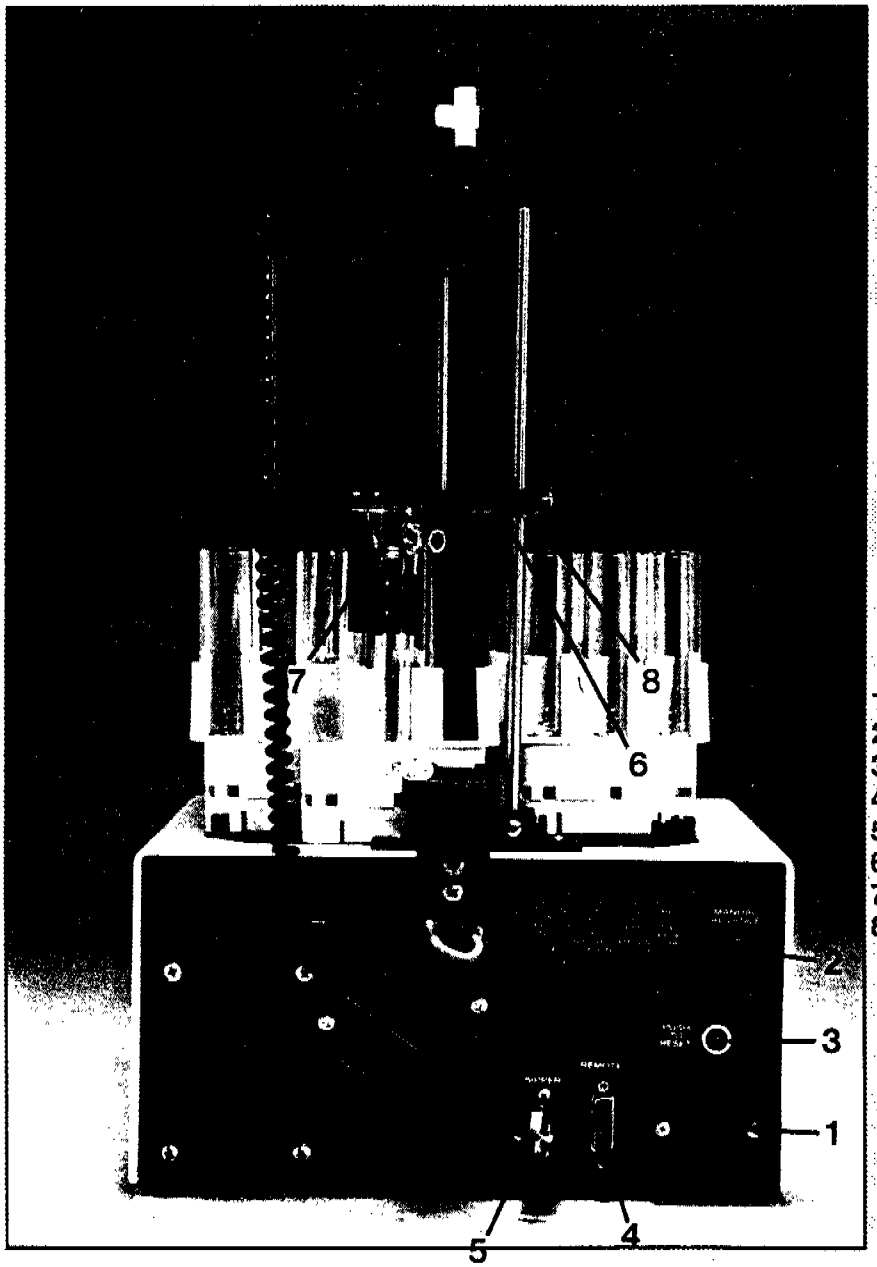
This button is used to reset the electronics of the ASM.

Remote Input Receptacle

This receptacle receives TTL level signals for operation of the ASM by the 4460A.

Sipper Output Receptacle

This receptacle provides electrical output necessary to move the **septum piercing needle** back and forth from the sample vial to the **wash vessel.**



Auto-sampling Module Rear View

1. ASM Power Receptacle
2. Manual Advance Button
3. Reset Button
4. Remote Input Receptacle
5. Sipper Output Receptacle
6. Hold-down Foot Assembly
7. Wash Vessel
8. Septum Piercing Needle Assembly

Hold-Down Foot Assembly

This assembly holds a sample vial in place as the **septum piercing needle** is lifted from inside it.

Wash Vessel

This vessel holds solution for rinsing the 4460A **purge vessel** between samples.

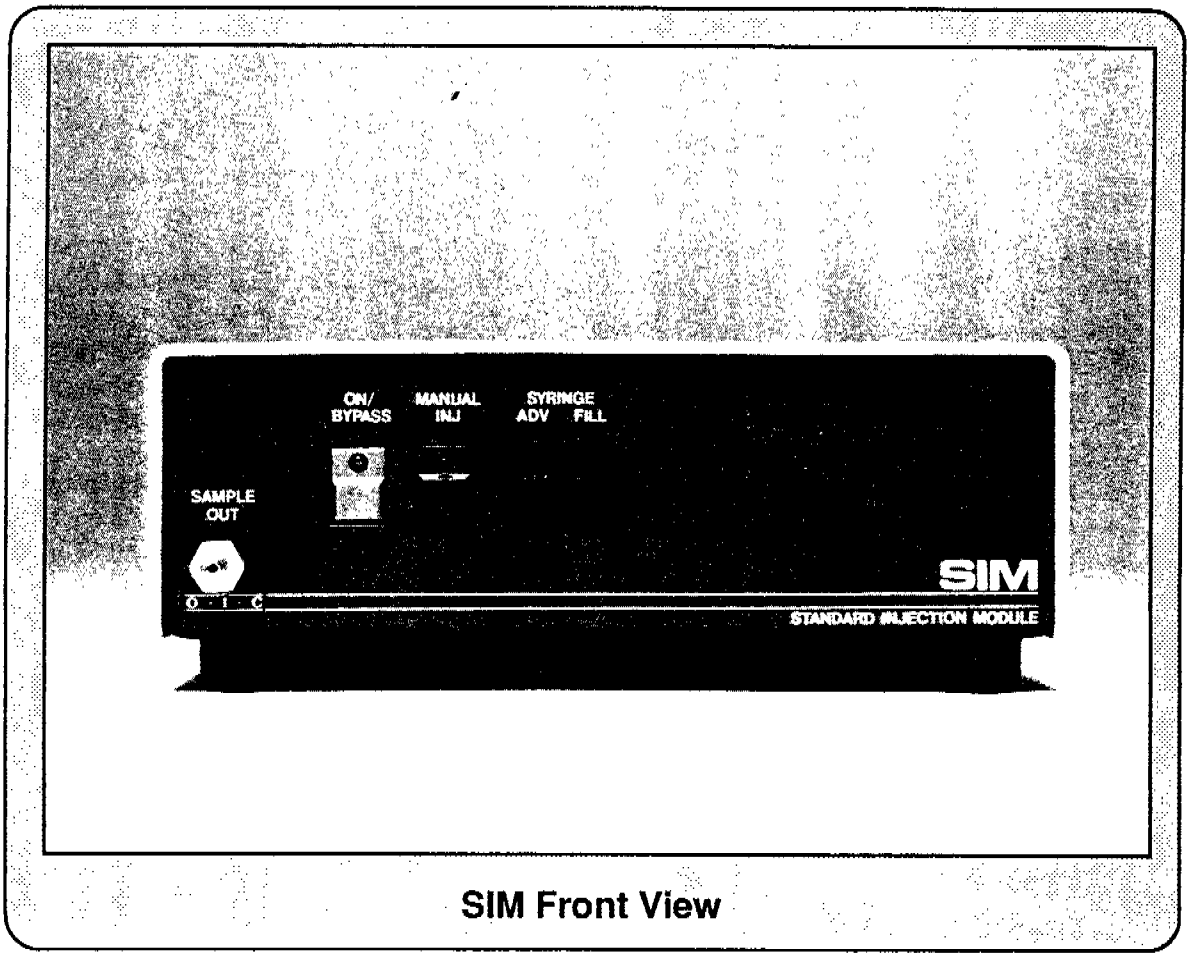
Septum Piercing Needle Assembly

This needle assembly consists of two concentric needles connected by a tee. The outer needle provides gas pressure to the sample vial to fill the **sample loop.** The sample travels from the vial through the inner needle into the sample loop.

Standards Injection Module (SIM) Front View

ON/BYPASS Switch

When this switch is ON (lamp is lit) the SIM is under the control of the 4460A Sample Concentrator in order to proceed through its automatic sequence. When the **ON/BYPASS switch** is on **BYPASS** (lamp is not lit) the automatic sequence signals



SIM Front View

from the 4460A are bypassed and the SIM may be operated manually by use of the other front panel switches.

MANUAL INJ Switch

When the **ON/BYPASS switch** is on **BYPASS**, this **MANUAL INJECT** switch can be used to inject a 10 µl aliquot of standard from the SIM into the sample stream between the 4460A and its autosampler. The injection sequence is comprised of (1) a valve movement to place the 10 µl valve bore in line with the standard solution flow path, (2) a syringe advance to flush 100 µl of standard through the valve bore, and (3) a reverse valve movement to place the captured 10 µl in line with the sample stream to the 4460A.

SYRINGE ADV Switch

When the **ON/BYPASS switch** is on **BYPASS**, pressing this **SYRINGE ADVANCE switch** causes the syringe to slowly advance, pushing its solution out. Continued holding of this switch speeds up the syringe movement and holding it down for more than two seconds causes the syringe to continue advancing even after the switch has been released. In this case, pressing the **SYRINGE FILL switch** stops the advance.

SYRINGE FILL Switch

When the **ON/BYPASS switch** is on **BYPASS**, pressing this **SYRINGE ADVANCE switch** causes the syringe to slowly retract, pulling in any solution connected to the syringe. In this way, the syringe may be filled without removing it. Continued holding of this switch speeds up the syringe movement and holding it down for more than two seconds causes the syringe to continue retracting even after the switch has been released. In this case, pressing the **SYRINGE FILL switch** stops the advance.

SAMPLE OUT Port

The sample and any injected standard passes through this port on its way to the **purge vessel** of the 4460A. A stainless steel line connects this port to the **LSM interface tee** of the 4460A.

Standards Injection Module (SIM) Side View

Standards Syringe

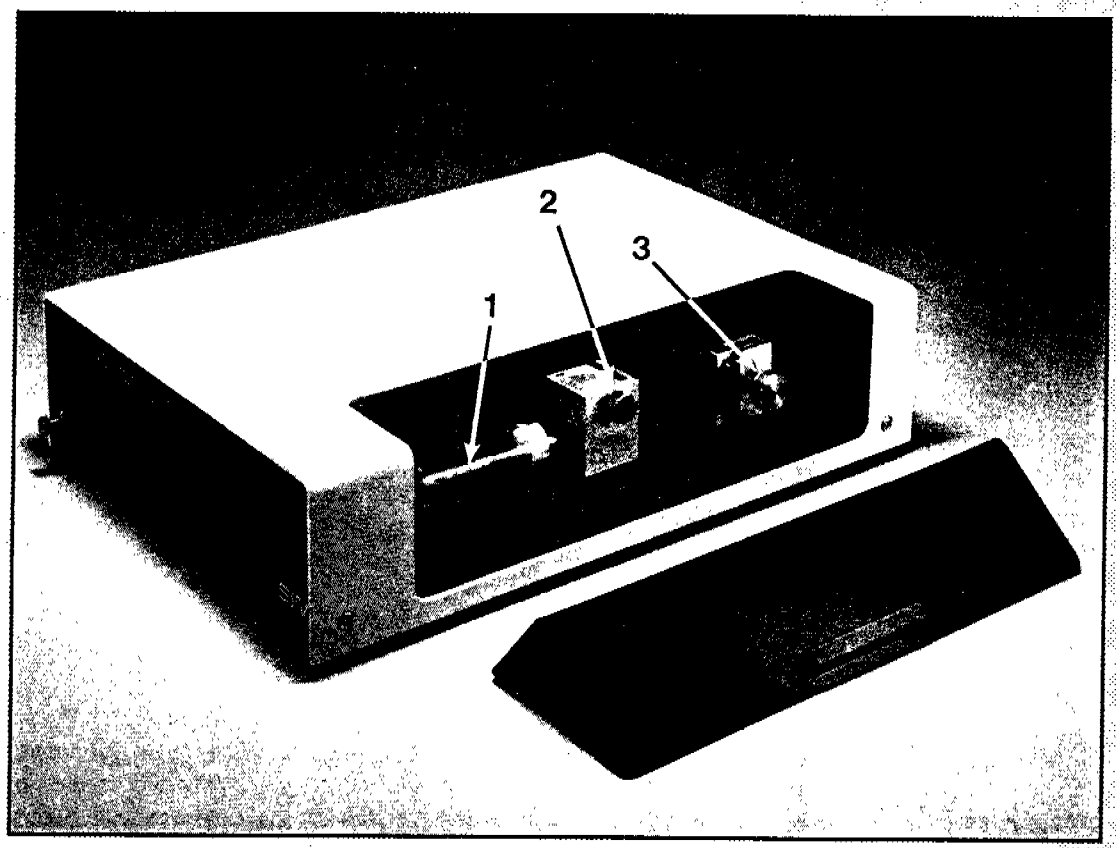
This 10 ml syringe contains the standard solution which fills the injection valve bore and is used for surrogate and internal standards addition.

Syringe Mounting Block

This block holds the **standards syringe** barrel in place. The syringe is removed by unscrewing the thumbscrew and allowing the outer block half to drop down.

Syringe Plunger Arm

This arm holds the syringe plunger as it moves back and forth during operation. It works to advance and retract the syringe plunger inside its barrel.



SIM Side View

1. **Standards Syringe**
2. **Syringe Mounting Block**
3. **Syringe Plunger Arm**

Standards Injection Module (SIM) Rear View

SPL IN Port

This port is connected to the **LSM sample output line.** Through it flows the sample to be purged on its way to the 4460A **purge vessel.** As the sample travels through the SIM, it has 10 µl of standard solution added to it.

SPL DRAIN Port

This port connects a waste line to your waste reservoir for unused standard solution. Solution exits here whenever the **standards syringe** advances. Likewise, solution can enter here to fill the syringe by placing the waste line into a vessel containing standard solution.

INJ MULTIPLIER Switch

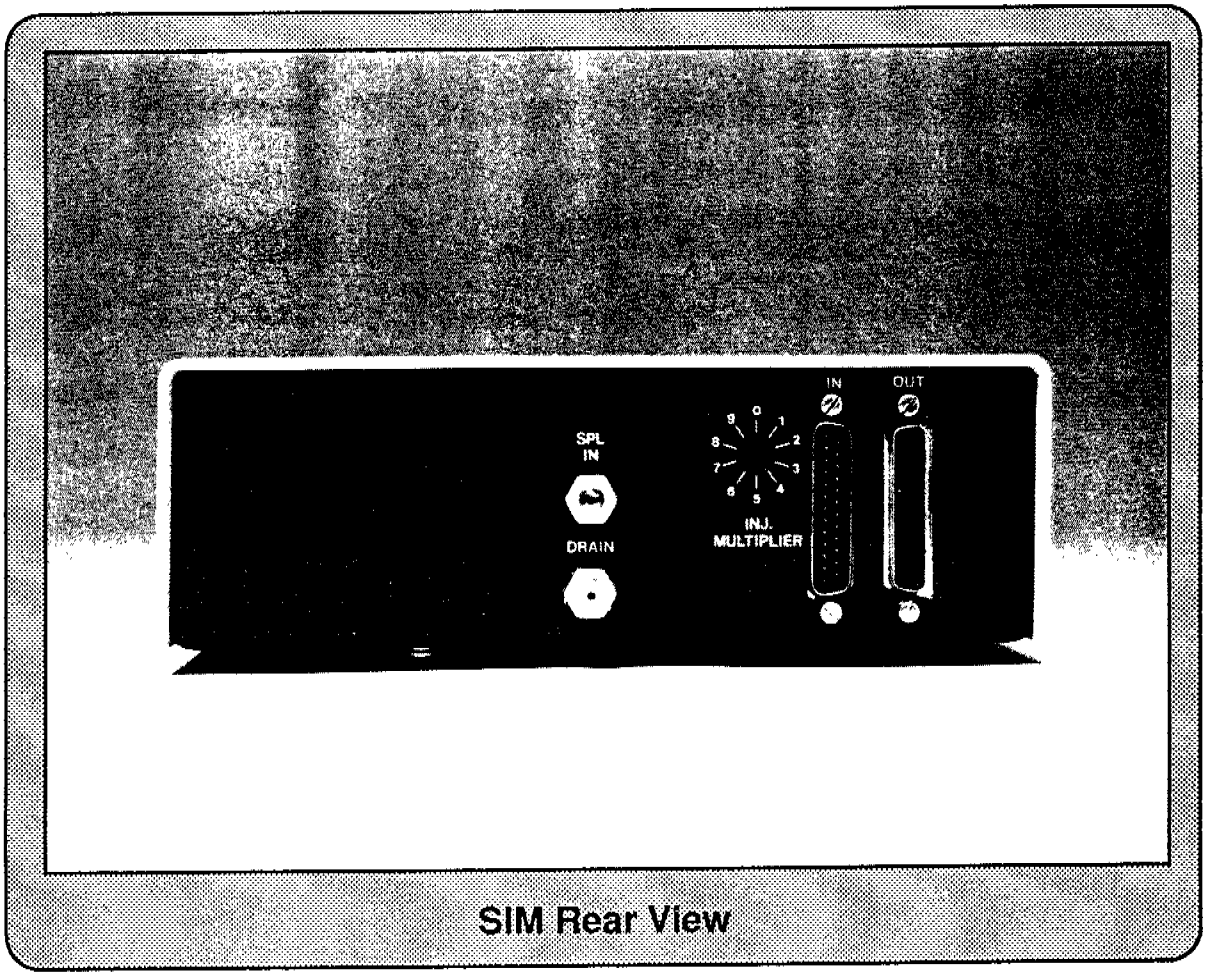
The number of 10 µl aliquots of standard solution which are injected into each sample as it flows through the SIM is set here. The **INJECTION MULTIPLIER switch** is set at the factory to setting 1, which injects one valve-bore of 10 µl, but can be set to inject as many as 9 valve-bores, or 90 µl.

IN Receptacle

This receptacle connects to the 4460A **autosampler cable receptacle** by using the cable provided. It brings 4460A control signals for SIM operation into the SIM electronic circuitry.

OUT Receptacle

This receptacle connects to the LSM control input receptacle by using the cable provided. It passes 4460A control signals from the SIM into the LSM and ASM.



SIM Rear View

Chapter 3
Installation

In **Chapter 2** the names and functions of the various components of the 4460A were outlined. Names of components were printed in boldface type to highlight them. These names are used here to refer to components involved in the installation of the instrument.

This chapter deals with the stepwise procedures used for properly installing the 4460A, its GC interfaces, and its optional autosampling system. The chapter begins by discussing materials needed for installation which are not included with the basic 4460A. The installer should gather the materials outlined here before attempting the 4460A installation, then proceed in a stepwise fashion through the instructions beginning with **Unpacking/Positioning the Instruments**, and referring to the installation figures.

Materials Needed

The following is a list of materials not provided with the basic 4460A but which are necessary for its installation:

Purge Gas Source

Refer to *Chapter 1 Specifications* for gas purity specifications.

A source of purge gas, regulated to a pressure between 30 and 250 psi (207-172 kPa) is needed for connection to the purge gas inlet. A hydrocarbon trap is provided with the 4460A for scrubbing contaminants from impure purge gas sources. External scrubbing of gas of questionable purity is highly recommended.

Carrier Gas Source

A source of carrier gas must be provided to the flow controller of the gas chromatograph (GC) interfaced to the 4460A. The purity and pressure requirements of this source should be specified by the manufacturer of the GC. For the 4460A, the purity requirement of this gas is the same as for the purge gas and the allowable pressure range is 0-250 psi (0-1724 kPa). This carrier gas may be provided from the same source as for the purge gas by using a tee fitting to split the gas supply line between the GC carrier inlet and the 4460A purge gas inlet. In this case, be sure that the regulated pressure is set at the source to a value within the allowable range of both devices.

Actuator Gas Source

A source of gas, regulated to a pressure between 40 and 80 psi (276-552 kPa) is needed for pneumatic actuation of the trap valve. Though neither high purity gas nor helium is needed here, this actuator gas may be provided from the same source as for the purge gas. This is recommended only if a single gas source is available. In this case, be sure that the regulated pressure is set at the source to a value within the allowable range of all components involved.

Gas Hookup Lines

A clean stainless steel or copper 1/8 inch (3.2 mm) OD gas hookup line is required to connect the purge gas source to the purge gas Inlet of the 4460A. A plastic or Teflon 1/8 inch (3.2 mm) OD x 1/16 inch ID (3.2 x 1.6 mm) gas hookup line is required to connect the actuator gas source to the actuator gas inlet. A Hookup Kit (OIC Part #177592) is offered for users who do not stock such tubing. This kit also includes an adaptor for connection of the metal tube to the purge gas regulator and a tee fitting for the actuator gas.

GC Interface Kit

A kit for the interfacing of the 4460A to the gas chromatograph is needed for connection of the GC carrier gas outlet to the 4460A carrier gas inlet and for the connection of the 4460A transfer line to the injection port of the GC.

Power Source

A standard receptacle for 115 VAC power capable of providing 8 amps, or 230 VAC at 4 amps, is required. The power cord is 6 ft (1.83 m) long.

4460A Setup

Unpacking/Positioning the Instrument

- Remove the Startup Kit and the 4460A from the shipping carton. Save all packing material until proper operation of the 4460A is verified.
- Place the 4460A next to the GC on the side nearest the GC injection port. The space under the 4460A should be kept clear of obstructions for proper cooling air flow during operation.

Gas Inlet Connections *(Refer to Figure 3.1)*

- Connect the purge gas line between the purge gas source and the purge gas inlet. (Tube nut and ferrule set are in the Startup Kit).
- Install the hydrocarbon trap onto the marked fittings on the rear panel.
- Connect the carrier gas line to the 4460A carrier gas inlet. This line is included in the GC Interface Kit. Connection of the other end of this line to the GC flow controller output is outlined later in this chapter.

- Connect the actuator gas line to the actuator gas inlet (must be 1/8 inch OD x 1/16 inch ID (3.2 x 1.6 mm) plastic or Teflon to be pushed over barb fitting). A length of clear urethane tubing is included in the Startup Kit for this purpose. Alternatively, the purge gas line can be teed as a source of actuator gas as shown in Figure 3.1.

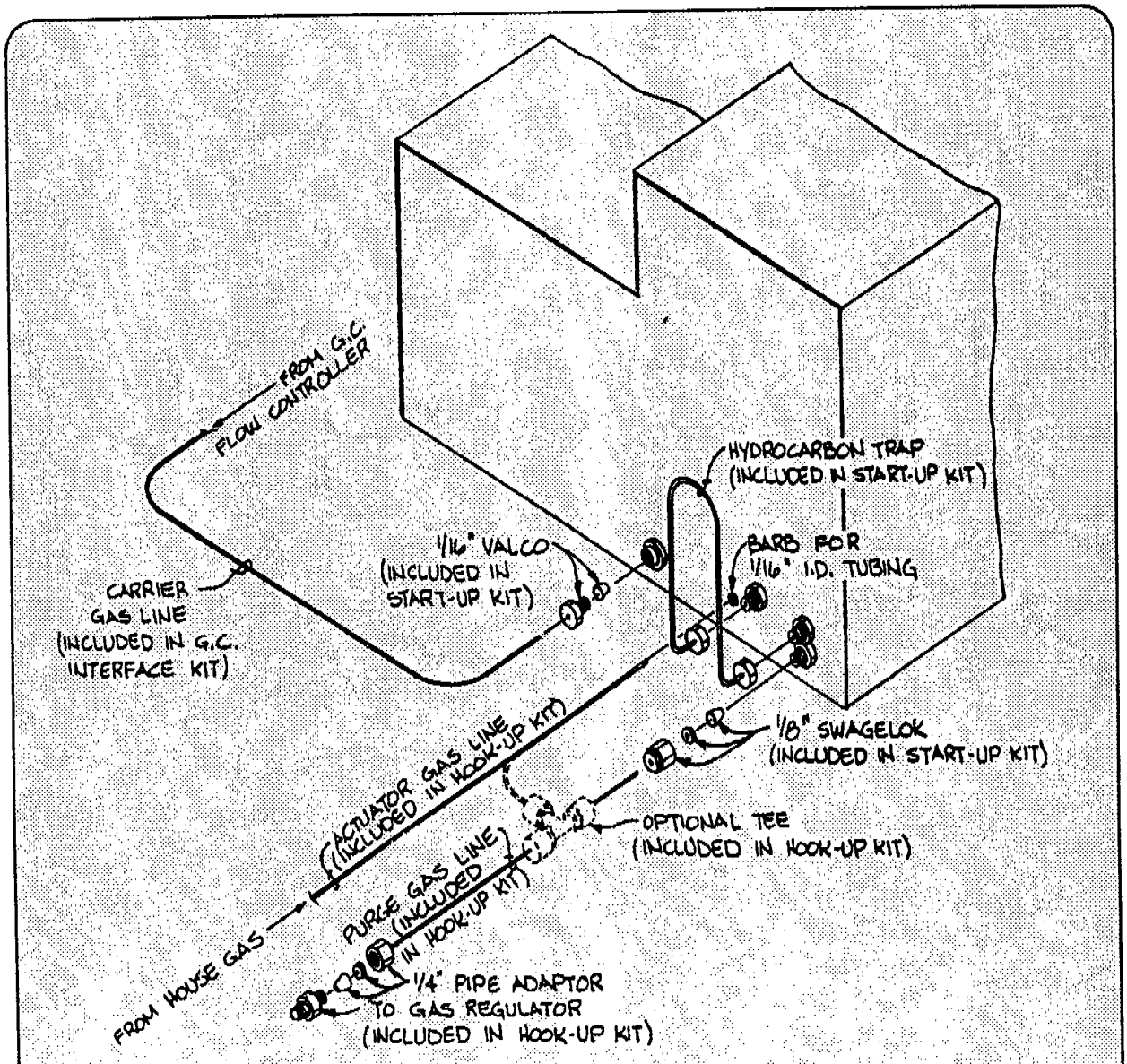


Figure 3.1 Gas Inlet Connections

Transfer Line and Drain Connections *(Refer to Figure 3.2)*

Port 5 of the trap valve may have a shipping plug in it, which must be removed. The free end of the transfer line is connected to the GC interface. These steps are outlined later in this chapter.

- Remove the left bay cover by pushing it rearward about 1/2 inch (1 cm) until it stops, then lift it upward to clear the 4460A.
- Remove the valve oven cover by turning the 1/4-turn latch to its vertical position, then pulling the cover outward.
- Connect the transfer line to the trap valve using the end of the line having the pre-installed nut and ferrule.
- Plug the transfer line thermocouple and power leads into their corresponding receptacles.
- Place the waste line from the bottom of the drain valve into a waste receptacle or route to a drain.

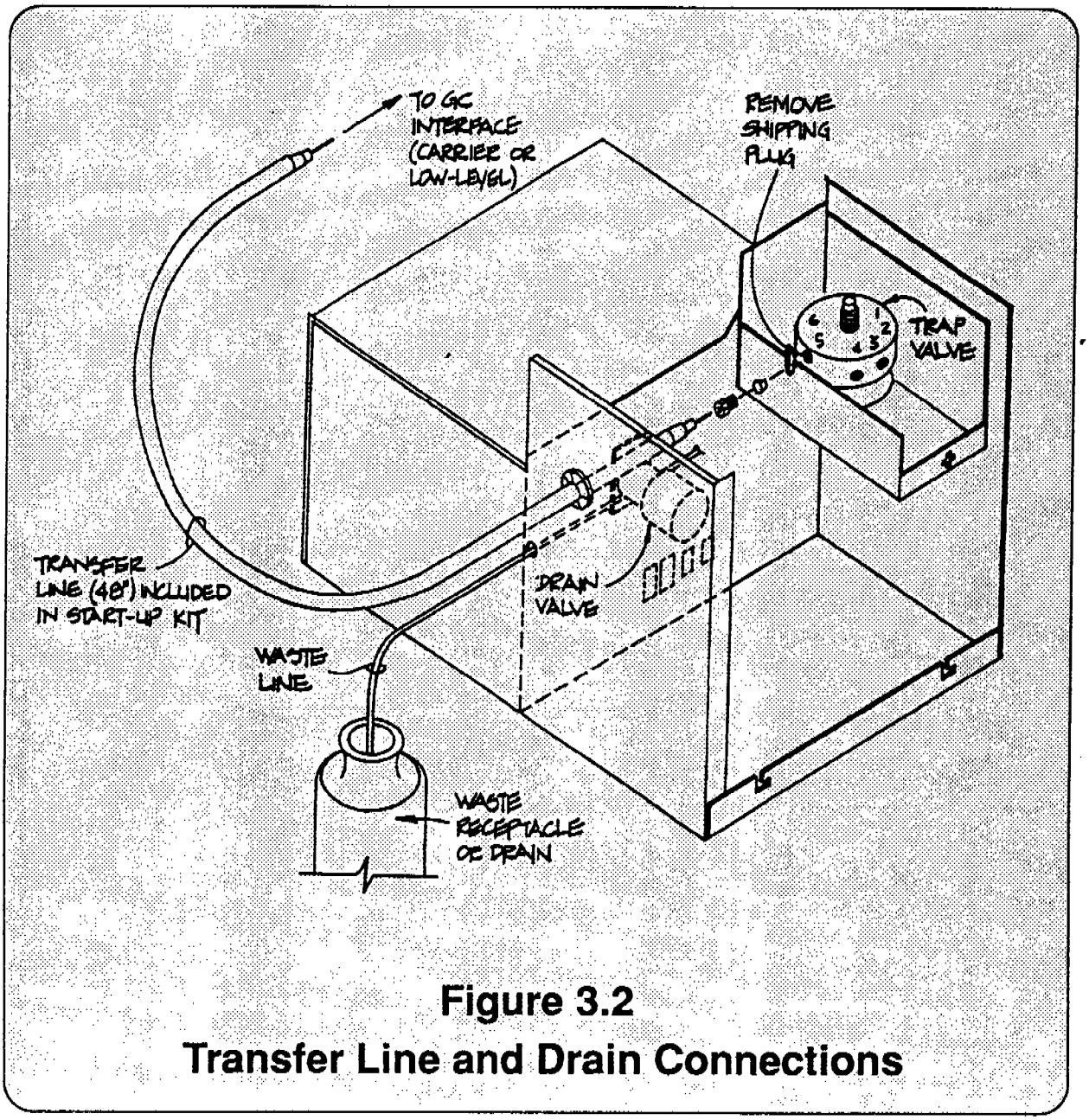


Figure 3.2
Transfer Line and Drain Connections

Sample Valve and Purge Vessel Assembly *(Refer to Figure 3.3)*

- Assemble the sample valve, syringe port, sample needle, and bracket.

- Install the sample valve/needle assembly onto the front panel as shown, but leave thumbscrews loose.

CAUTION
Be sure that the top ferrule, through which the needle passes, is in place in the tee fitting.

- Remove the bottom nut and ferrule from the tee fitting and slide them onto the purge vessel top tube (note the orientation shown in Figure 3.3). Insert the purge vessel top tube into the tee fitting while inserting the purge line down into the septum plug in the purge vessel side arm. While insuring that the purge vessel top tube is fully inserted into the tee, gently tighten the bottom nut to form a leak-tight seal.

CAUTION
Over-tightening the drain line may deform the tube flare or strip the tube nut threads.

- Adjust the position of the sample valve/needle assembly so that the needle touches the purge vessel frit without bowing. Tighten the thumbscrews to fix this position.

- Tighten the top nut of the tee fitting to form a leak-tight seal between the sample needle and the tee.

- Screw the drain line directly into the side port of the sample valve. Tighten to form a leak-tight seal.

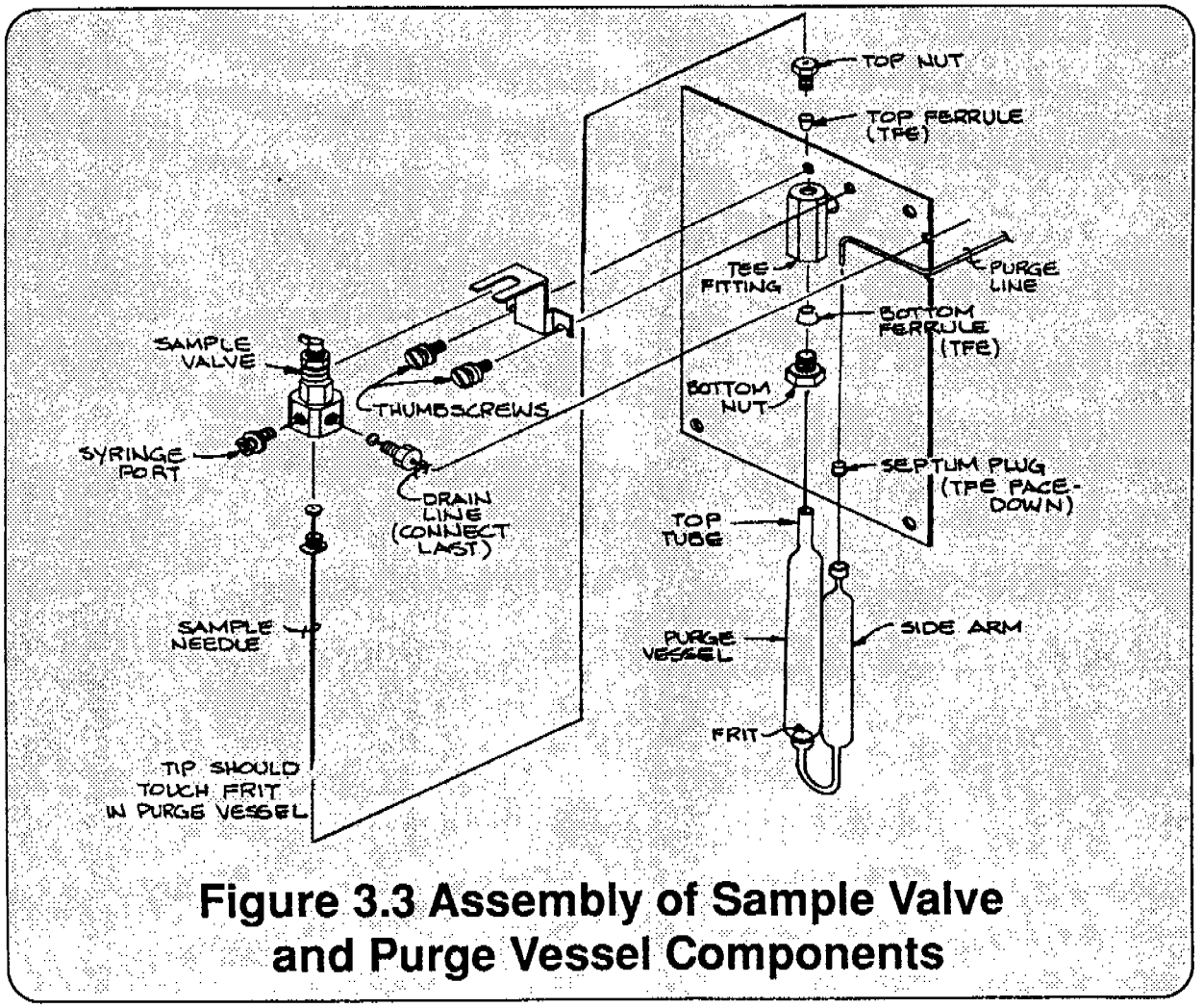


Figure 3.3 Assembly of Sample Valve and Purge Vessel Components

Trap Installation *(Refer to Figure 3.4)*

A detailed description of traps and various available adsorbent packings is found in Chapter 2.

- A #3 trap (OV-1/Tenax/Silica Gel) is installed in the 4460A when shipped from the factory. These instructions are provided for the changing of traps. *The 4460A should be powered down when changing traps.* The valve oven cover need not be removed.

CAUTION
Do not heat a newly-installed trap before flushing it with purge gas.

- While supporting each trap bulkhead fitting with a 7/16 in. open-end wrench, disconnect each trap leg with a 3/8 in. open-end wrench.
- Unplug the trap thermocouple and remove the trap. Place yellow dust caps on the trap removed from the 4460A.
- Plug the new trap thermocouple into its receptacle.

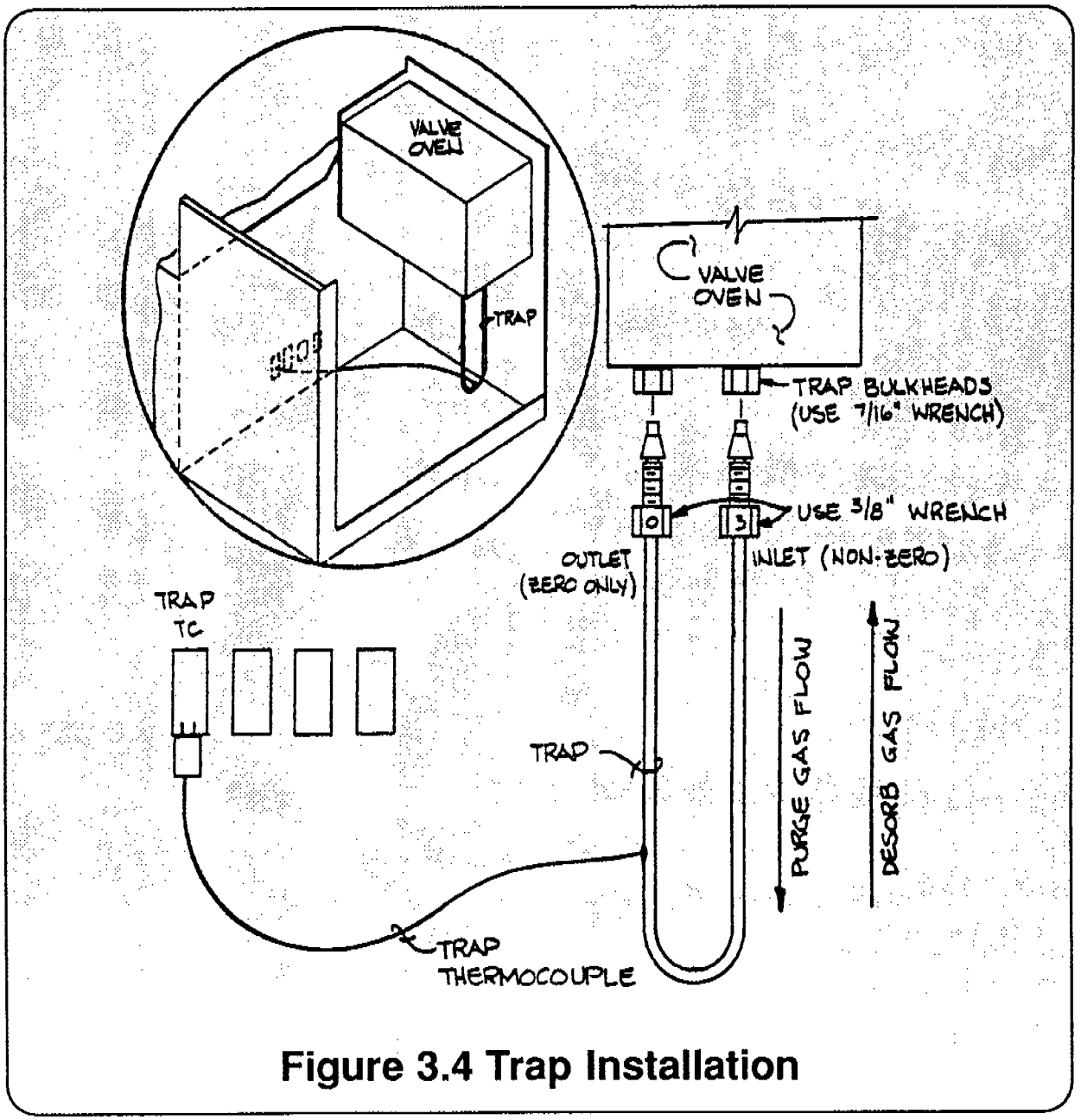


Figure 3.4 Trap Installation

- In like manner, connect trap legs from the new trap to the bulkheads. The trap leg stamped with a non-zero number is the *inlet* and should go toward the front of the 4460A.

Standard Carrier Interface Connections *(Refer to Figure 3.5)*

See ***Chapter 1, GC Interface Kits*** *for a description of the concept and application of the Carrier Interface Kits. These kits are to be used with packed column injectors.*

Parts named in this section which are part of the gas chromatograph include the flow controller, injector, injector body, and heater block.

- Locate the carrier gas flow controller output line in the gas chromatograph. Disconnect this line from the flow controller outlet port or from the output of any hydrocarbon trap mounted downstream of the flow controller.

- Connect the 4460A carrier gas line to the flow controller (or hydrocarbon trap) outlet port using the 1/8 in. Swagelok nut and ferrule set on the end of the

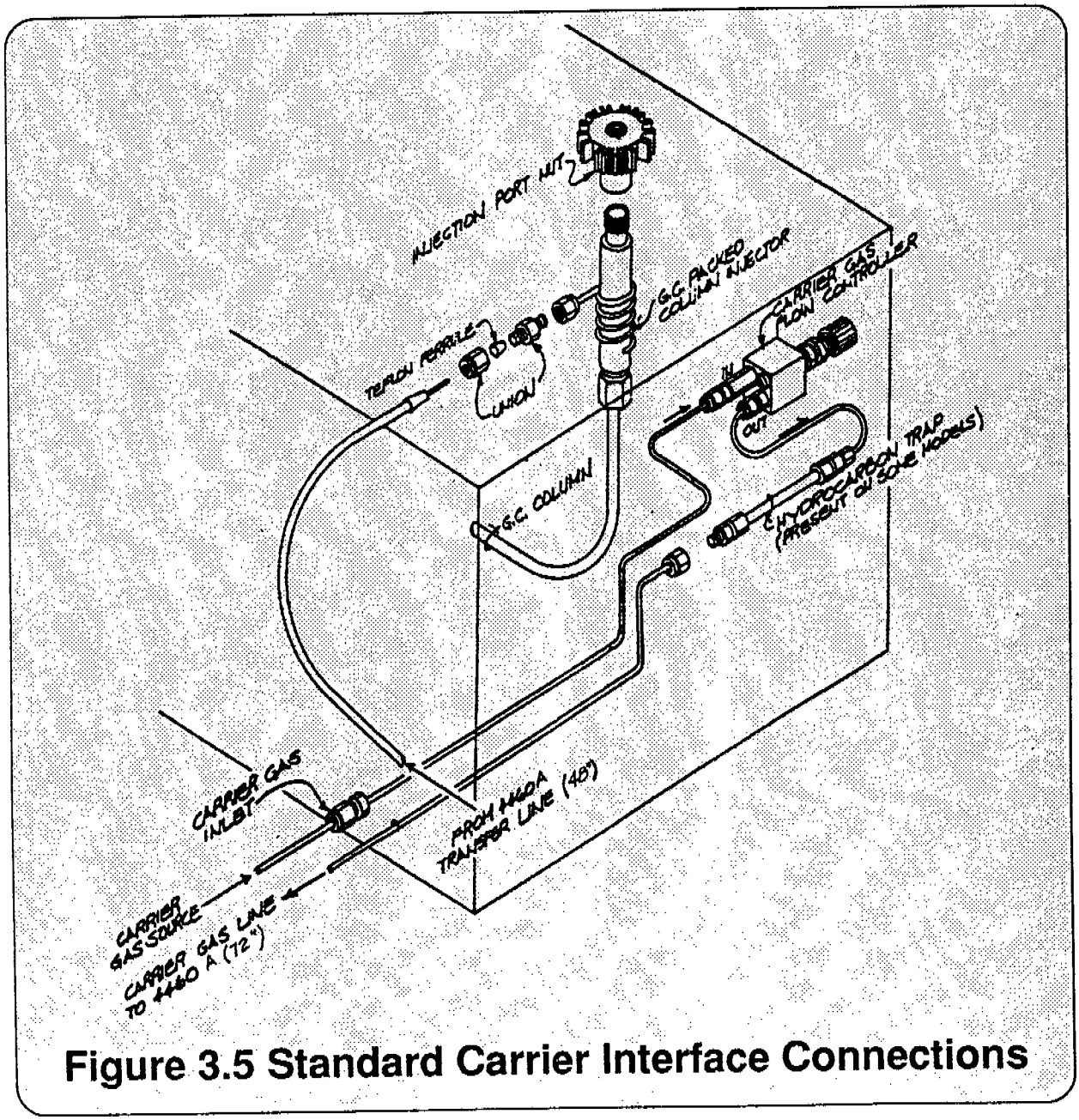


Figure 3.5 Standard Carrier Interface Connections

tube. The other end of this line should be connected to the 4460A carrier gas inlet as outlined earlier in this chapter.

- Remove the GC packed column Injector if necessary, to coil the line disconnected from the flow controller outlet around the injector body or heater block. Coil tightly, then re-secure the injector.

  The purpose of coiling is to keep the tubing hot, using the injector heater block. This coiled line will become part of the overall sample transfer conduit to the column and should be kept at a temperature at least that of the 4460A transfer line. The union which connects these two lines should also be kept in the heated zone adjacent to the injector heater block.

- Connect the free end of the coiled line to the 4460A transfer line using the union and ferrule supplied in the carrier interface kit. Use the Teflon ferrule to seal the transfer line end to the union if permanent swaging of a metal nut and ferrule on this line is not desired. Be sure to seat each line in the bottom of the union to minimize dead volume.

- Install column, injector septum, adaptors, carrier gas inlet line, etc., as specified in the GC manual. The GC can still be operated normally (the injector can be used for head-of-column injections) without disconnecting the 4460A interface lines.

Low-Dead-Volume Interface Connections *(Refer to Figure 3.6)*

See Chapter 1, GC Interface Kits for a description of the concept and application of the Low-Dead-Volume Interface Kits. These kits are to be used with packed column injectors, even though fused silica and glass capillary columns are used.

Parts named in this section which are part of the gas chromatograph include the injector, injector body, injector septum nut, adaptor nut, adaptor ferrule, and flow controller. The capillary column is purchased separately.

- Locate the carrier gas flow controller output line in the gas chromatograph. Disconnect this line from the flow controller outlet port or from the output of any hydrocarbon trap mounted downstream of the flow controller.

- Connect the 4460A carrier gas line to the flow controller (or hydrocarbon trap) outlet port using the 1/8 in. Swagelok nut and ferrule set on the end of the tube. The other end of this line should be connected to the 4460A carrier gas inlet as outlined earlier in this chapter.

- Remove the injector septum nut and septum from the GC packed column injector. Replace with the column injection kit assembly, spacer, and support nut from the interface kit as shown.

- Insert the column adaptor into the injector body from below (inside the oven) until the column injection kit assembly tube (1/16 in. OD) protrudes from the bottom of the adaptor. Gently tighten the adaptor nut to hold the column adaptor in place.

- Place the graphite ferrule (OIC Part #165523) from the column injection kit over the protruding tube, then follow it with the union from the kit. Screw these two pieces into the bottom of the column adaptor until fingertight.

- Refer to Figure 3.6 Detail. Slide the column adaptor up or down inside the injector body until the protruding 1/16 in. OD tube is aligned with the bottom of the union. Tighten the union to seal the tube against it, then tighten the adaptor nut to secure the column adaptor.

The adaptor nut (with adaptor ferrule) seal against the column adaptor is only for mechanical support. It does function as a gas-tight seal.

- Depending on the diameter of the capillary column to be connected, select a column ferrule from the column injection kit based on the following table:

Column ID	Ferrule ID	OIC Part #
0.22 mm	0.4 mm	178509
0.32 mm	0.5 mm	178491
0.53 mm	0.8 mm	178483

- Insert the capillary column end through the column nut and column ferrule as shown.

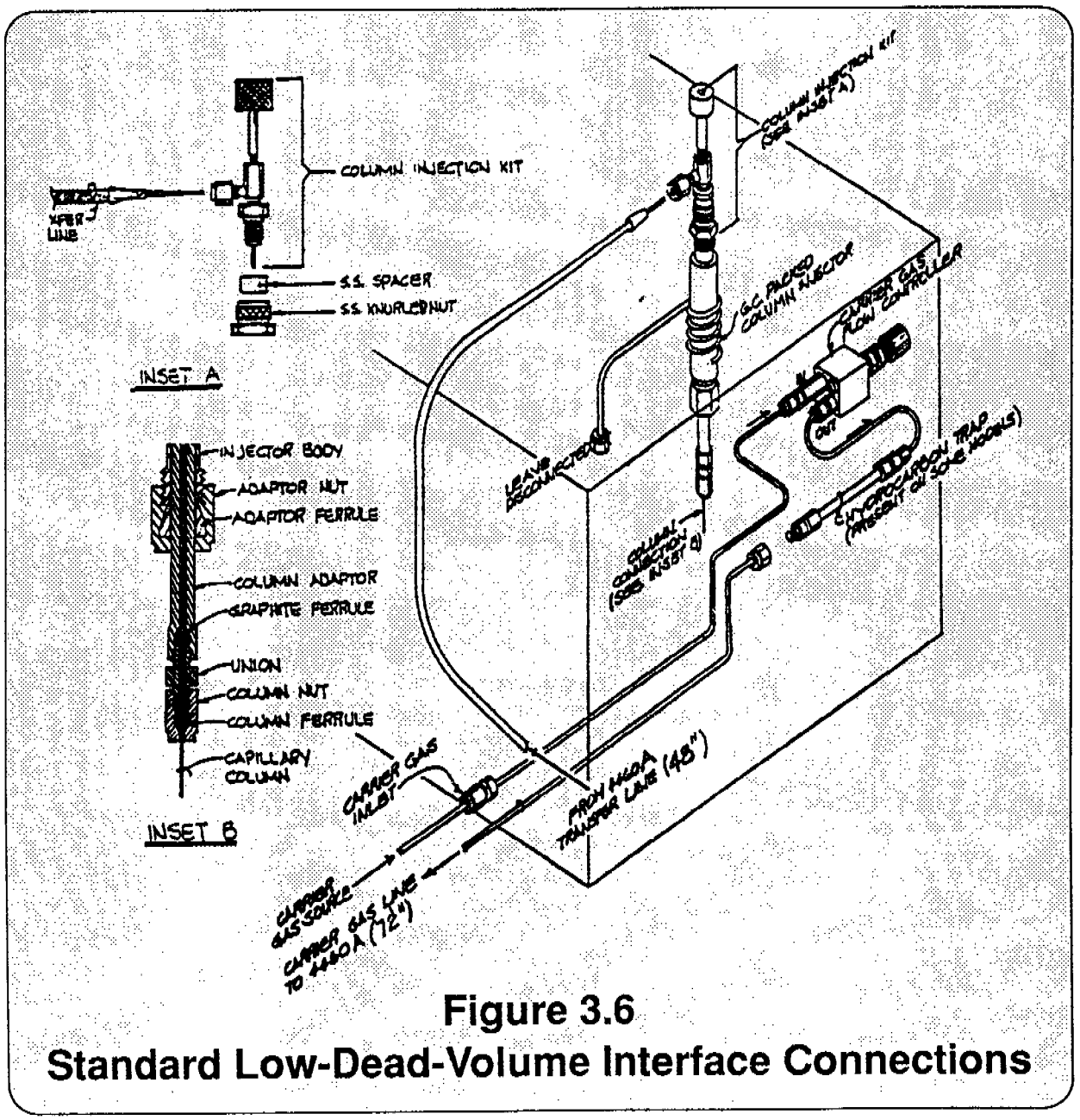


Figure 3.6
Standard Low-Dead-Volume Interface Connections

- Score and break the column using standard capillary handling procedures to preclude any ferrule particles lodged in the end of the column from entering the system. The column end should not be pointed upward during the breaking step to keep fragments from falling into the column. Refer also to the specific instructions provided with the column.

- Unscrew the tee with its needle guide from the injector body, revealing the 1/16 in. OD guide tube. Carefully insert the end of the column up into the column injection kit assembly tube, until it lifts the guide tube up and confirm that the column lodges inside the guide tube. Press the guide tube and column back down, then tighten the column nut to form a seal between the column and the tube. Reinstall the tee and internal septum (Teflon face down), then tighten the needle guide in place.

Electrical Connections *(Refer to Figure 3.7)*

*Refer to **Chapter 2** for discussion(s) of the uses of GC oven temperature feedback, the Start DESORB Input, and the DESORB Relay Output.*

- If GC oven temperature feedback is desired, plug the GC thermocouple into its receptacle and insert the thermocouple into the GC oven.

- If a GC-ready input signal from the GC to the 4460A is desired to start DESORB and this option is available, connect the GC-ready input cable from the GC to Terminals 6 and 7 of the Terminal Strip-Relay I/O. This cable may be ordered separately from OIC where applicable to the GC. The GC supplier may also supply it. To use this GC-ready input signal to start DESORB, DIP switch #7 should be off.

- If remote start of the GC column program and integrator are desired at the beginning or end of DESORB and these options are available, connect remote-start output cables from the GC and the integrator to terminals 4 and 5 of the Terminal Strip-Relay I/O. These cables may be ordered separately from OIC where applicable to the GC. The GC or integrator supplier may also supply them.

- Remove the right bay cover by pushing it rearward, then upward (the left bay cover must be removed first) if the voltage select plug needs to be changed from 115 VAC to 230 VAC.

- Replace bay covers.

- Remove the power cord from the Startup Kit and plug it into the main AC power receptacle, then connect power cord to line voltage.

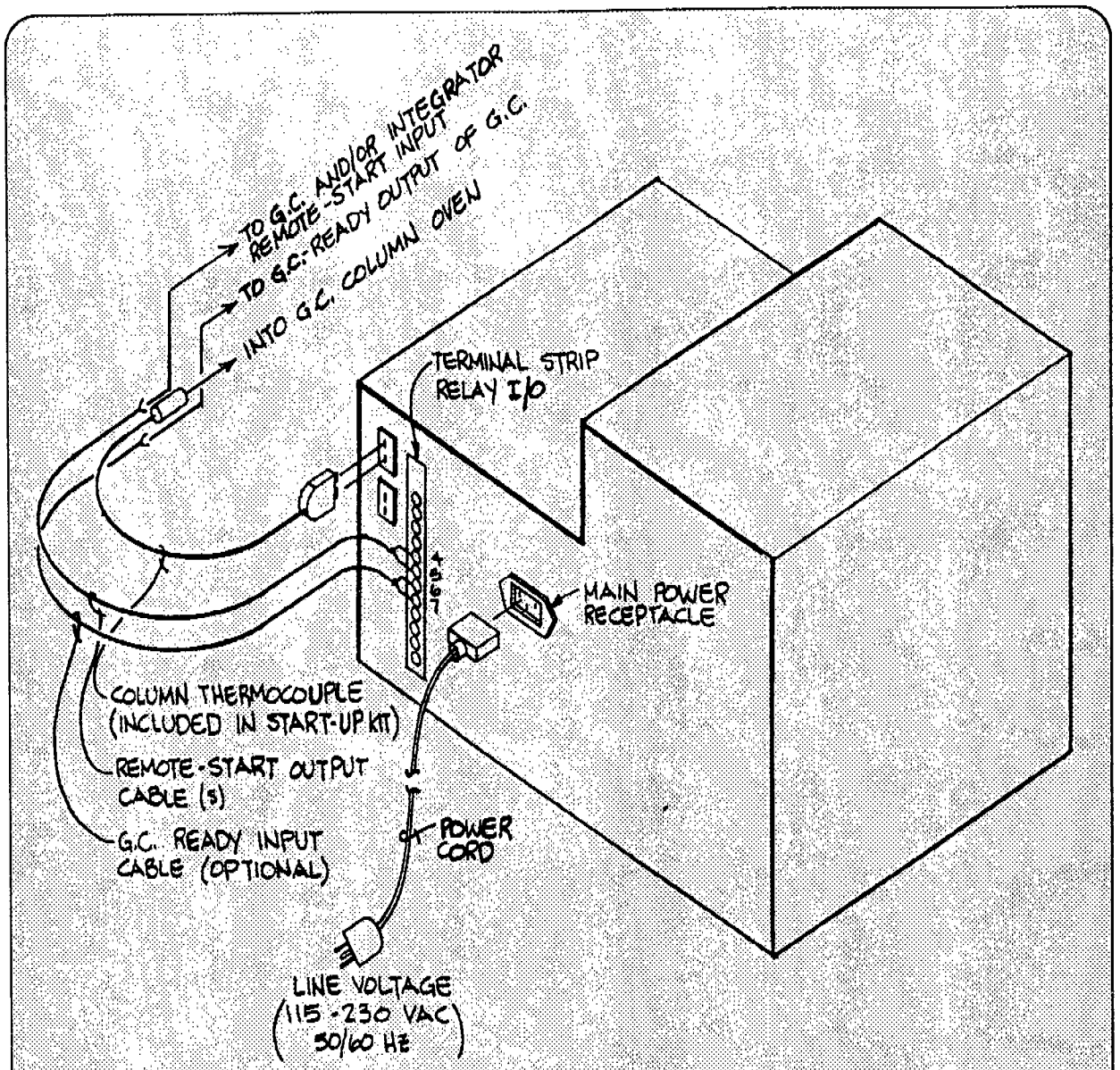


Figure 3.7 Electrical Connections

Autosampler System Setup

Autosampling Module (ASM) Assembly *(Refer to Figure 3.8)*

The ASM is part of the optional 4460A Autosampling System. It is one of two modules included with this option, the other being the Loop Sampling Module (LSM). Detailed instructions concerning the ASM can be found in the separate Autosampler Instruction Manual provided with each ASM. This Original Equipment Mfrs (OEM) manual describes various configurations of the ASM. Configurations used by the 4460A Autosampling System are found in section 2.2C (Sampler with Wash Station Assy) and section 2.9 (30 mm and 100 mm septum piercing accessory for Units with Wash Station). The 4460A ASM Septum Piercing Needle Assembly is somewhat different from the one shown in the OEM manual. Refer to the separate manual only if these instructions do not fully answer your question.

CAUTION:
The wash station can be connected to a flowing stream as well as a gravity feed rinse. Be sure that potential spillage would not drip onto electrical connections on the back of the ASM.

- Screw the threaded pipet rod into its guide on the ASM.
- For standard (40 ml vial) systems, slide the spacer over the pipet rod and adjust it until it seats firmly at the bottom of the guide.
- Slide the wash vessel into its bracket on the wash station assembly, then slide the assembly over the pipet arm and into place resting on the spacer.
- Slide the actuator arm assembly over the two protruding rods as shown, and secure in place using its lock screw. Plug its electrical connector into the ASM connector marked "SIPPER".
- Install the septum piercing needle assembly onto the actuator arm assembly.

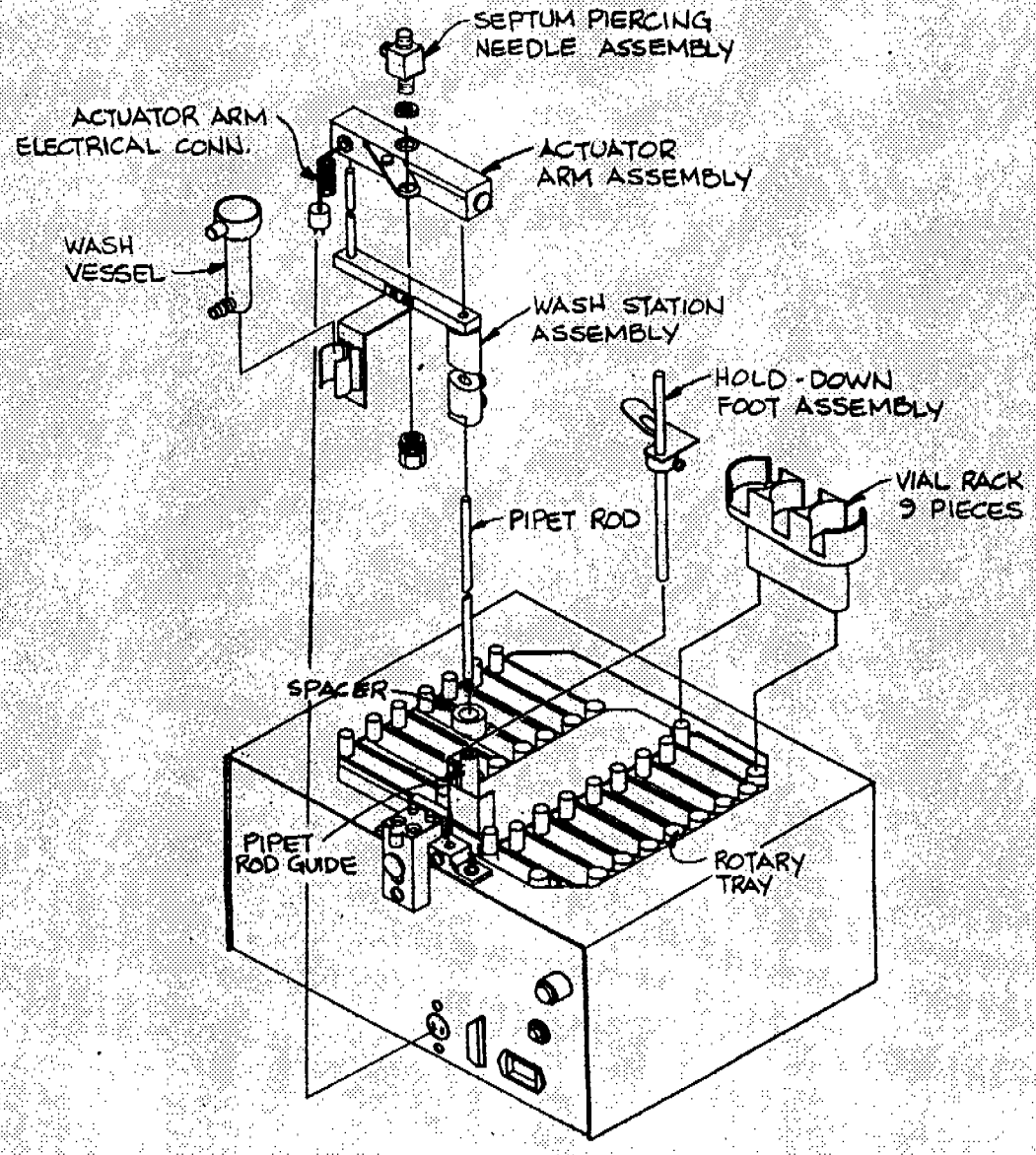


Figure 3.8 Autosampling Module (ASM) Assembly

- Insert the hold-down foot assembly into its mounting hole, align the hole in the foot with the septum piercing needle, and secure the rod using its lockscrew.

- Press vial racks (set of 9) onto *every other rack position* on the rotating tray. The red rack can be used as a manual last-sample rack, past which the rotary tray will not advance. The 4460A has the ability to set the number of samples, however, and will stop the rotary tray at the specified sample number, so the use of the red vial rack is optional.

Tubing Connections *(Refer to Figure 3.9)*

- Arrange 4460A, ASM, and LSM as shown.

- Disconnect the 4460A drain line from the sample valve, connect the LSM

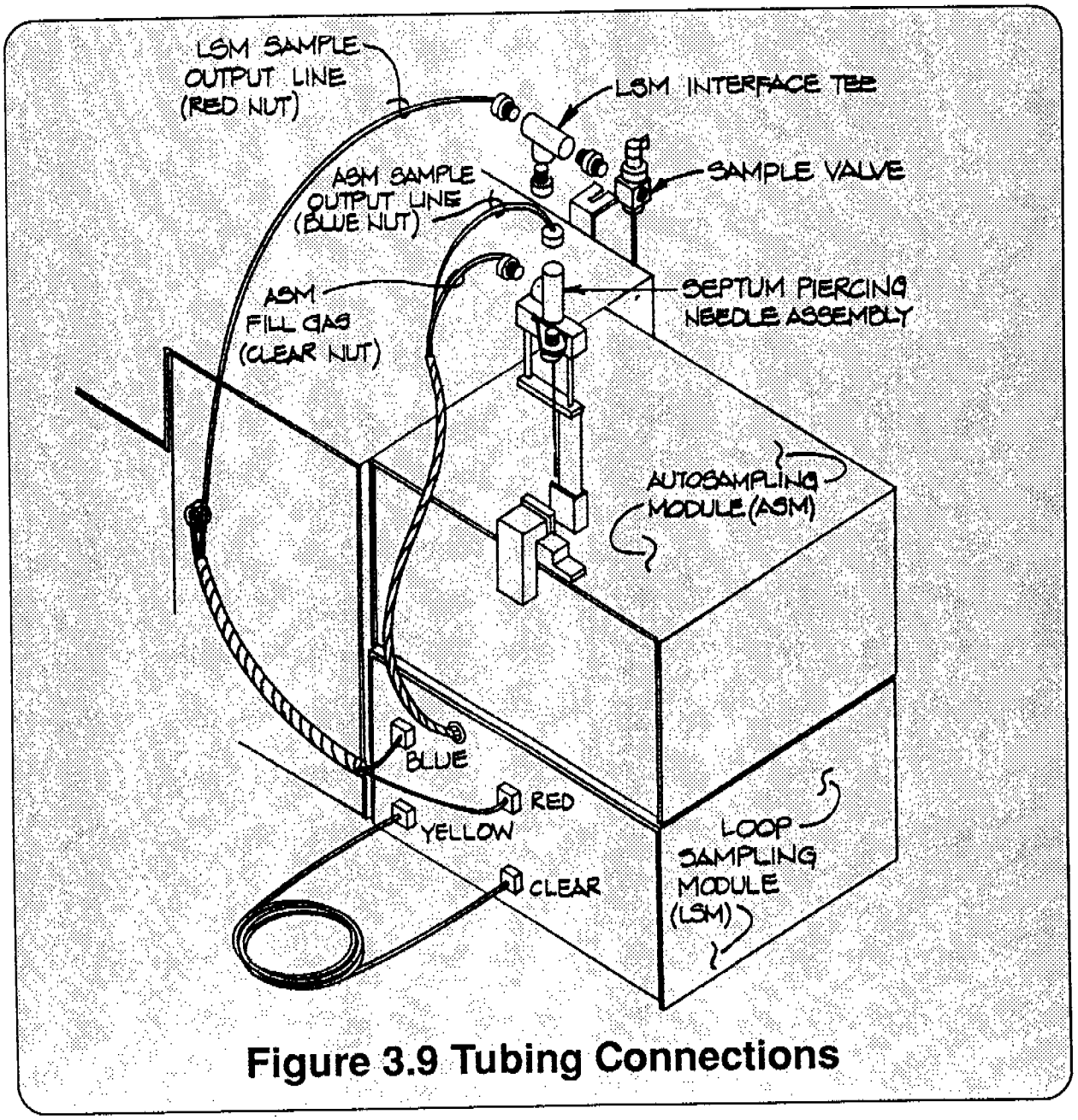


Figure 3.9 Tubing Connections

The 4460A and the Autosampling Module (ASM) should be assembled before attempting these tubing connections.

interface tee in its place, and reconnect the drain line to the bottom of the tee.

- Connect the LSM fill gas line to the autosampler gas outlet on the 4460A.
- Route the LSM sample outlet line (red nut) through the 4460A drain line feedthrough, through the front panel notch, and connect to the remaining port of the LSM interface tee.
- The LSM fill gas outlet line (clear nut) and LSM sample inlet line (blue nut) are bundled. Connect them as shown to the septum piercing needle assembly (blue to top port; clear to side port of tee).

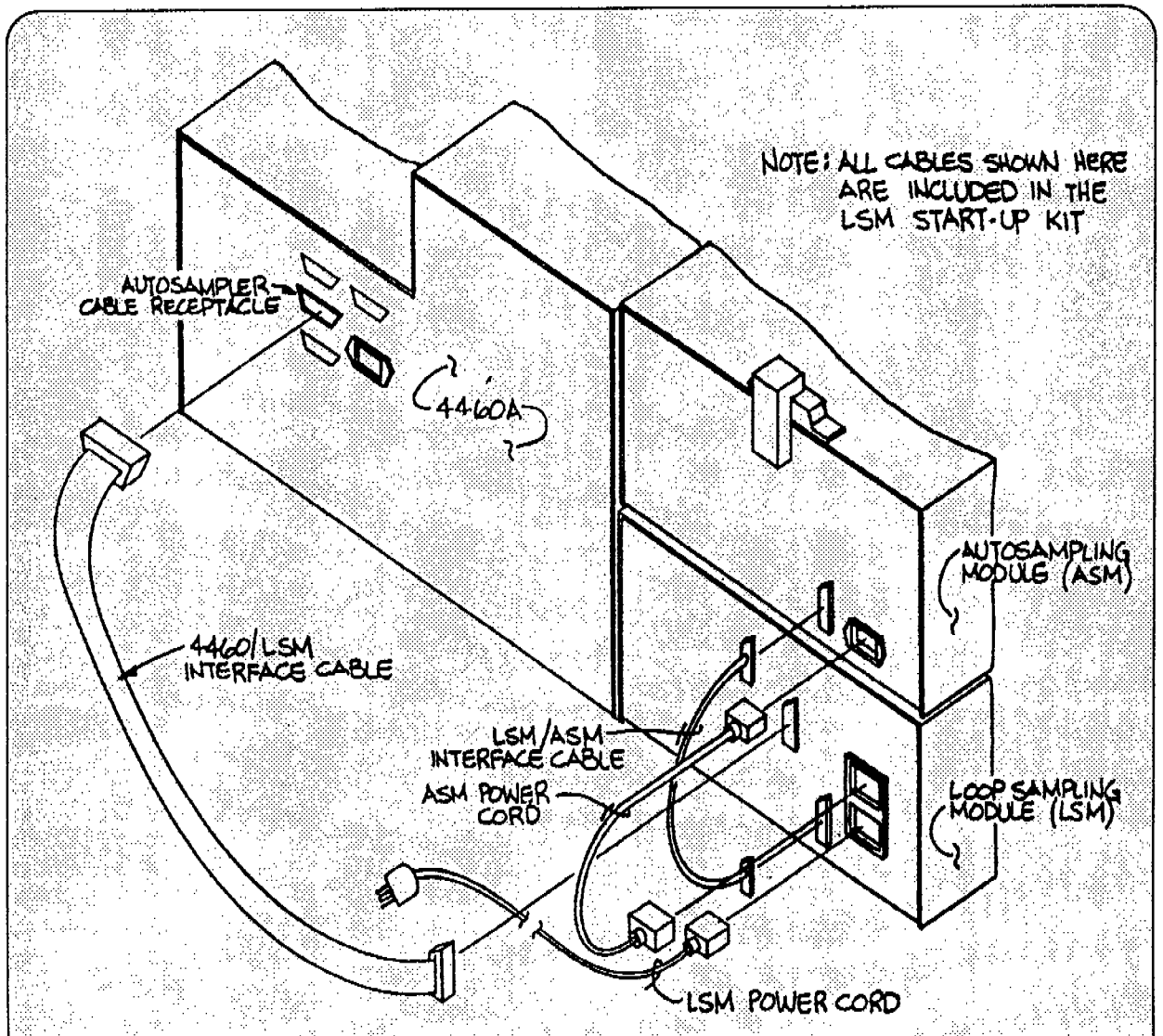


Figure 3.10
Autosampling System Electrical Connections

Electrical Connections *(Refer to Figure 3.10)*

Complete Tubing Connections before attempting these electrical connections.

- Connect the 4460A/LSM interface cable between the 4460A and the Loop Sampling Module.
- Connect the LSM/ASM interface cable between the Loop Sampling Module and the Autosampling Module.

The ASM gets its power from the LSM.

- Connect the ASM power cord between the Autosampling Module and the Loop Sampling Module.
- Connect the LSM power cord to the Loop Sampling Module, then connect the other end line voltage (115 VAC).

Setup of SIM with 4460A and LSM

Tubing Connections *(Refer to Figure 3.9)*

- Place the SIM on top of the 4460A right bay.
- Connect the Teflon drain line to the drain port using the nut and ferrule provided.
- Connect the new LSM sample output line provided with the SIM between the LSM and the SPL IN port of the SIM.
- Connect the short stainless steel tube provided with the SIM between the SPL OUT port of the SIM and the LSM interface tee on the 4460A front panel.

Electrical Connections *(Refer to Figure 3.10)*

- Unplug the 4460A/LSM interface cable from the 4460A and replug it into the SIM OUT receptacle.
- Plug the new cable included with the SIM between the 4460A autosampler cable receptacle and the SIM IN receptacle.
- Plug the power supply module output cable into its mating receptacle on the back

Needle Sparging Setup

Needle Sparging Assembly (Refer to Figures 3.11)

A 5 ml or 25 ml needle sparging kit is available as an option for the 4460A. Either of these options allows for purge and trap analysis to be performed on appropriate size samples of matrices such as soils, sludges or other solids. Needle sparging may also be beneficial for liquids that foam. Samples may be purged at ambient temperature or at elevated temperatures as required by some EPA method using an optional sparging vessel heater.

If the 4460A is set up for fritted sparging, remove the septum plug, purge vessel, bottom nut, bottom ferrule, top ferrule, top nut and sample needle (Refer to Figure 3.3) by performing the reverse procedure given for sample valve and purge vessel assembly earlier in this chapter.

- Assemble the sample valve with the sparging needle.
- Slide the top nut and 1/16 in. top ferrules onto the sparging needle as shown in Detail 1.
- Install the sample valve/needle assembly onto the front panel as shown, but leave thumbscrews loose.
- Assemble the 1/2 x 1/4 in. reducing union on the neck of the needle sparging vessel.
- Slide 1/4 in. TFE ferrule on the 1/4 in. OD gold plated tube.
- Insert the 1/4 in. tube and ferrule fully into the tee fitting and secure it by screwing the reducing union in place. While ensuring that the purge vessel neck is completly inserted into the tee and union, tighten first the union into the tee and then the bottom nut around the neck of the sparging vessel. Both of these fittings should be made fingertight only.
- Adjust the position of the sample valve/needle assembly so that the needle touches the bottom of the purge vessel. Tighten thumbscrews to fix the position.
- Slide the flangeless nut and Kel-F ferrule onto the needle sparging adaptor tube as shown in Detail 2 and screw it into the sampling valve as shown fingertight.
- Install a 1/16 in. stainless steel nut and TFE ring ferrule on each 4460A purge line and the sparge adaptor tube and tighten them down on the stainless steel union with a 1/4 in. wrench as shown in Detail 3.

There is not Auto Drain capability with needle sparging and the 4460A drain tube is left disconnected for this type of analysis

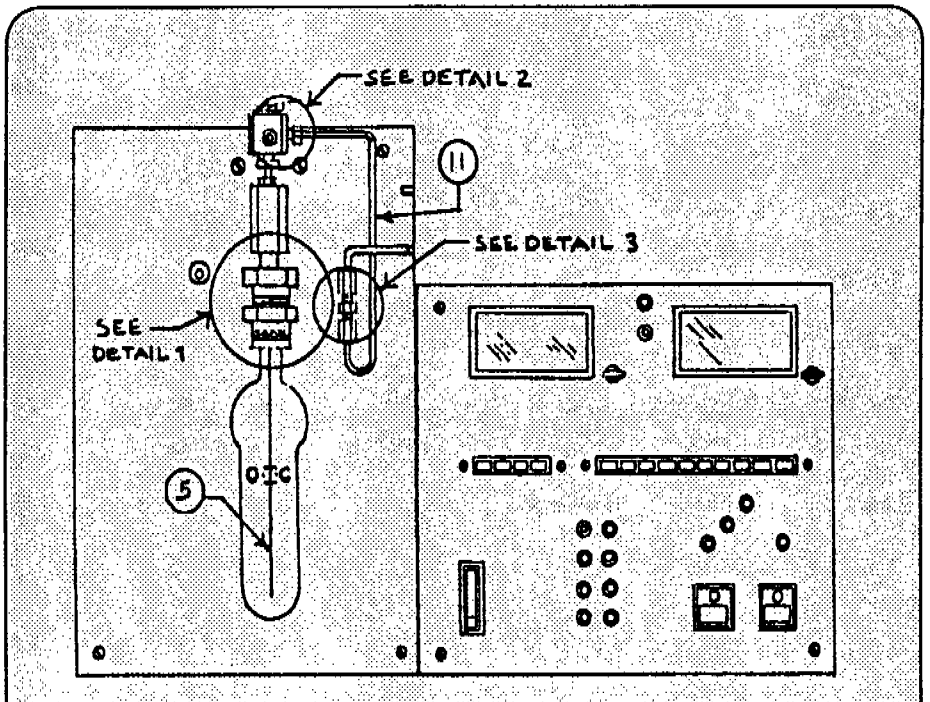


Figure 3.11 Needle Sparging Connections

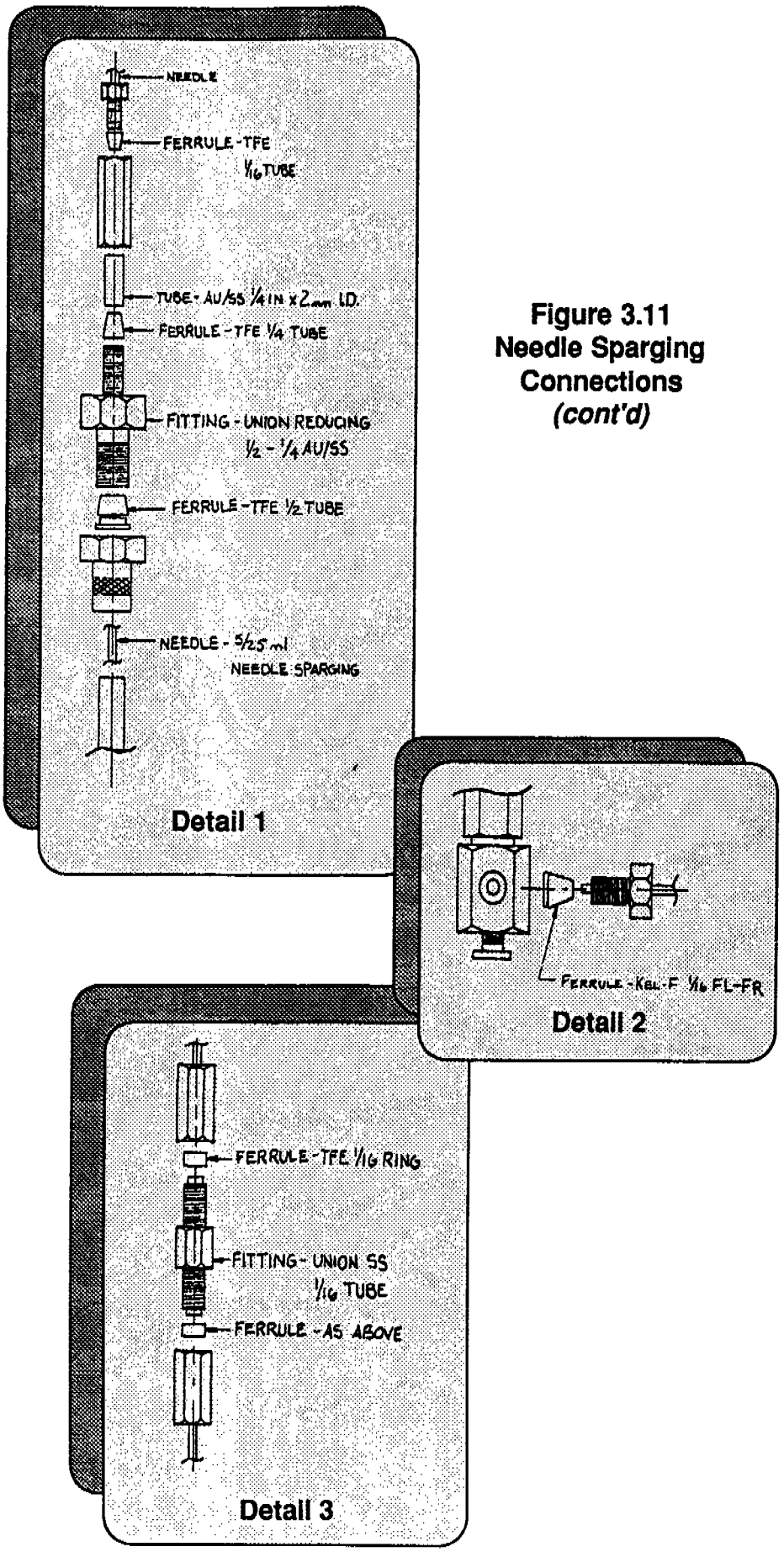


Figure 3.11
Needle Sparging Connections
(cont'd)

Chapter 4
Operation

In the last chapter, installation of the 4460A and its optional autosampling system is described. This chapter deals with the operation of the equipment for analyzing samples. The instructions here assume that the procedures outlined in **Chapter 3, Installation** have been completed. This chapter begins with a summary of the methods used by the 4460A for sample analysis, including diagrams of the flow paths in each state. Then a pre-operational checklist is outlined to confirm proper setup and function, followed by step-by-step procedures for running a sample. The sectioncalled **4460A Functional Checks** is to be performed before the first time the unit is operated, and can also be used to spot-check the instrument's function on a daily or routine basis. This section outlines steps to confirm proper function; it does not give step-by-step instructions on how to diagnose or fix a problem. Diagnostic steps to take in case one of these functional checks is not confirmed are given in **Chapter 5, Maintenance and Troubleshooting.**

Overview of Method

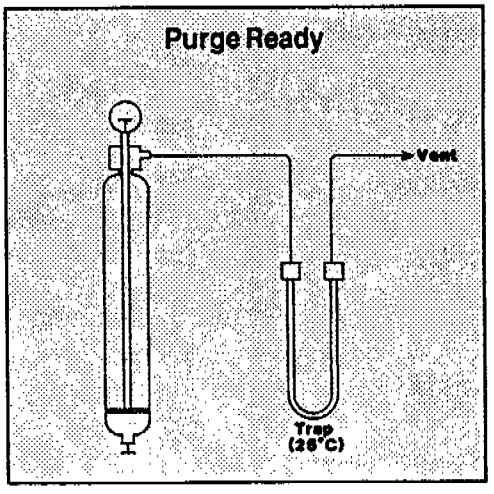
Sample Introduction

A water sample may be introduced into the instrument either by syringe injection or means of a calibrated sample loop. The loop is used with the autosampler and for automatic sampling from a bottle or a flowing process stream. The sample loop also affords greater consistency of injection volume. Standards or extracts in solvents may also be injected directly onto the column for studies of trap and column efficiencies.

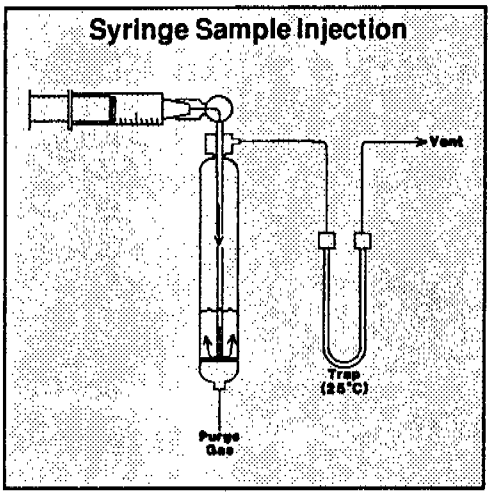
In order for you to understand the steps of operation in running a sample, diagrams of the 4460A flow paths during a sequence are shown next. The first set of diagrams shows the sequence of analysis when injecting a sample with a syringe. The second set shows an autosampler injection.

4460A Syringe Injection

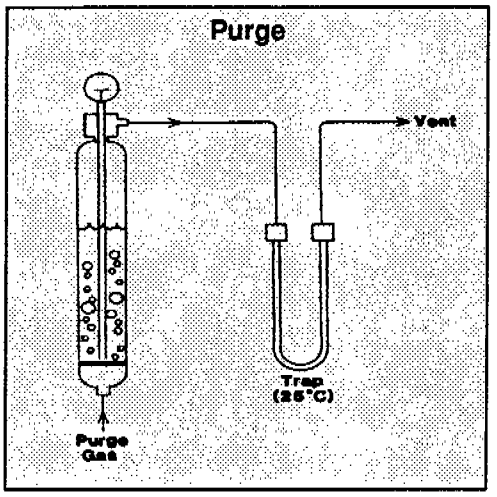
The system advances from STANDBY to PURGE READY when the trap has cooled to the Purge Temp setpoint.



The system can then be advanced to PURGE, the sample injected...

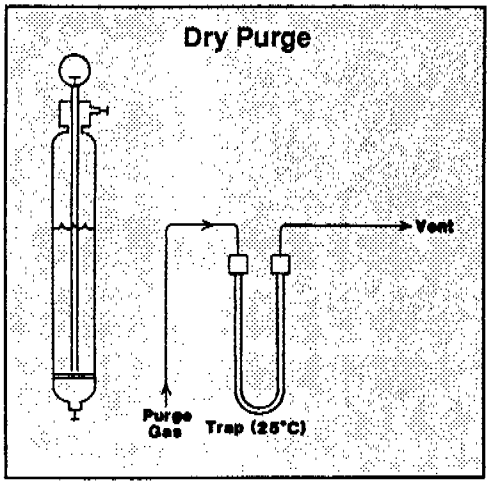


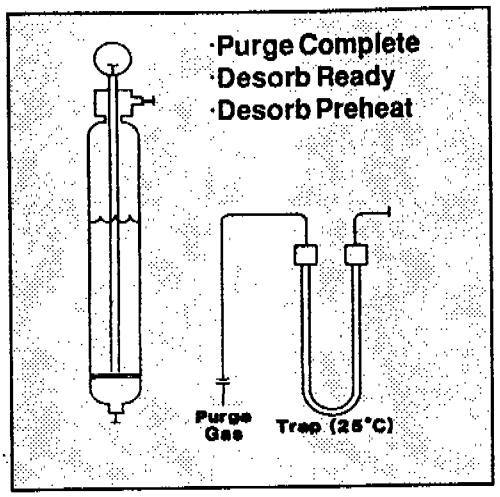
...and purging/trapping of the components from the water sample begins.



In most methods, this DRY PURGE time is set to 00:01

When the purge time is complete, the system advances to DRY PURGE, and the purge gas bypasses the purge tube to carry dry gas through the trap until the DRY PURGE timer reaches its pre-set time.



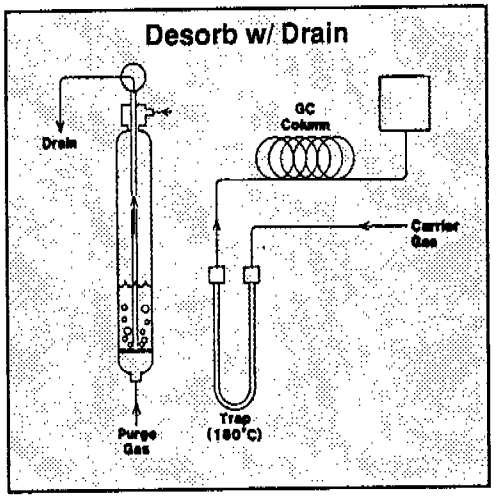


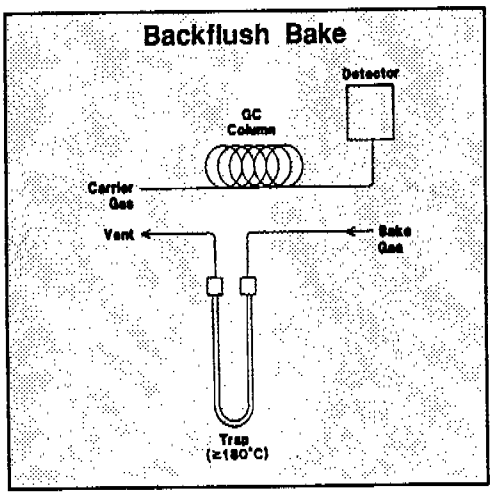
The system will then advance to PURGE COMPLETE and hold the sample on the trap until the GC oven temperature decreases to the pre-set Column Temp setpoint (as after a temperature-programmed GC analysis). The system will then advance to DESORB READY until a GC-ready signal is received from the gas chromatograph (this state can be skipped).

The PREHEAT step is typically not needed for good compound resolution using the 4460A. The default setpoint is 20° C so that this state is passed without any trap heating before DESORB.

Once the GC oven is ready to accept the sample, the system advances to DESORB PREHEAT and the trap is rapidly heated without gas flow.

When the trap temperature reaches the Desorb Preheat Temp setpoint, the system advances to DESORB W/DRAIN, the trap valve is actuated, and the trapped components are plug-desorbed onto the head of the GC column.





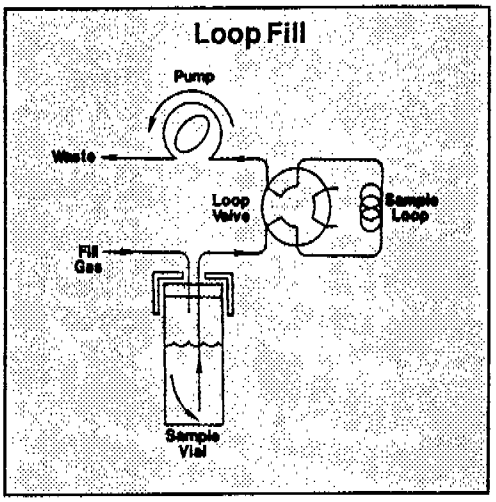
The trap temperature is held at the Desorb Temp setpoint until the Desorb Time setpoint is reached. The system then advances to BACKFLUSH BAKE.

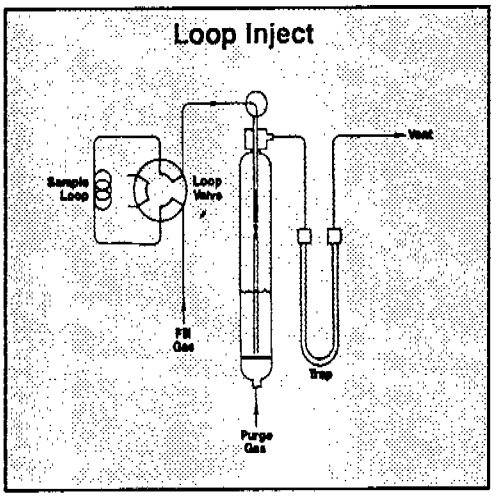
In BAKE, the trap temperature is maintained at the Bake Temp setpoint until the Bake Time setpoint is reached. The system then advances to STANDBY, the trap cools back to the Purge Temp setpoint, and is ready for the next sample when PURGE READY is indicated.

Autosampler Injection

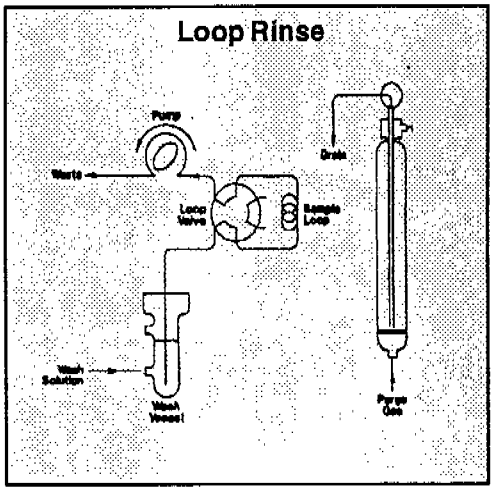
The system advances from STANDBY to PURGE READY when the trap has cooled to the Purge Temp setpoint.

It then advances immediately to LOOP FILL, where the sample vial is pierced and pressurized. Loop fill gas pushes the sample from the bottom of the vial, through the sample loop, and against the restriction of the running peristaltic pump. The pump acts to meter a constant rate of sample through the sample loop and keeps the loop volume and vial under pressure.





When loop fill time is complete, the system advances to LOOP INJECT, where the loop volume is pushed by purge gas pressure into the purge vessel. The inject time is adjusted so that purge flow continues for a few seconds after sample transfer to insure completeness. The flow rate of the purge gas in this state is the same as in the PURGE state.



The system then sequences through the primary states of operation until the end of DESORB W/DRAIN. Before advancing to BAKE, the system goes through two rinses with wash solution. Wash solution (water or an alcohol solution) is drawn from the wash vessel through the sample loop to waste by the pump. This rinse time can be set to achieve short or long rinses. The wash solution is then injected into the purge vessel in a manner identical to the sample then drained, and the rinse procedure is repeated. The system then advances to BAKE and resumes the primary sequence.

4460A Functional Checks

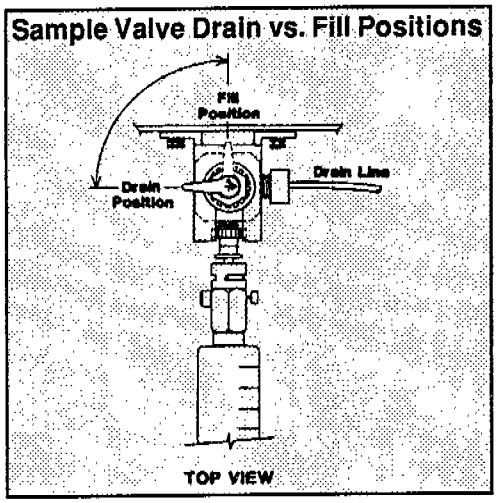
Here are some step-by-step procedures for confirming the proper function of the 4460A before sample analysis. Refer to **Chapter 5, 4460A Diagnostic Tools** for explanations of how each of these checks can be used to isolate problems.

Pre-Power-Up Checks

- Turn on purge, carrier, and actuator gas pressures.
- Confirm that carrier gas flow is set to desired rate.
- Refer to Chapter 2, Dip Switches if necessary, to set DIP switches to desired settings concerning:
  - Enabling of autosampler sequence (switch #1)
  - Default method settings (switches #2, 3 and 4)
  - Relay closure for GC program and integrator starts (switch #5)
  - Auto-advance past PURGE READY (switch #6) and for DESORB READY (switch #7)

CAUTION:
If the handle arrow is not truly aligned with the drain position, sample draining will be slow or non-functional.

- Point arrow on sample valve handle to Drain position (see figure at right).
- Confirm that the rear panel auto drain switch is set to ON (set toward the near edge of the instrument).
- Turn on power switch. Internal neon lamp should glow when power is on. Default method designator should appear temporarily on the Temperature display.



These Functional Checks can also be used for troubleshooting. Read *Chapter 5, 4460A Diagnostic Tools* in order to interpret symptoms caused by various problems.

Confirming Trap Valve Rotation

- Remove both instrument covers and the oven cover to expose the trap valve.
- Press the manual button on the trap valve actuator solenoid (mounted on the rear panel) while watching the trap valve. The valve rotor should rotate 90° clockwise (viewing from above) when actuated.
- Find the stop-pin on the bottom of the trap valve but still inside the oven. Press the manual actuation button again and confirm full travel of the stop-pin against its stop in both directions of rotation. If the pin does not rest against its stop in both directions, the valve actuator should be realigned to prevent a restriction or stoppage of flow due to the valve ports not being fully aligned. Call our Service Department for assistance if this is the case.

Setting Purge Gas Flow Rate *(Refer to Figure 4.1)*

Confirm that the purge gas flow is set to the desired rate (40 ml/min is specified for most methods) by the following steps:

- While the system is in STANDBY or PURGE READY, set the internal purge gas pressure regulator to read 20 psi, as indicated on the pressure gauge.
- Press the front panel AUTO-ON/OFF key to turn OFF (internal lamp should go off).
- Press RUN/STOP-STEP key to advance to PURGE state. State status is indicated by the STATE STATUS LEDs.

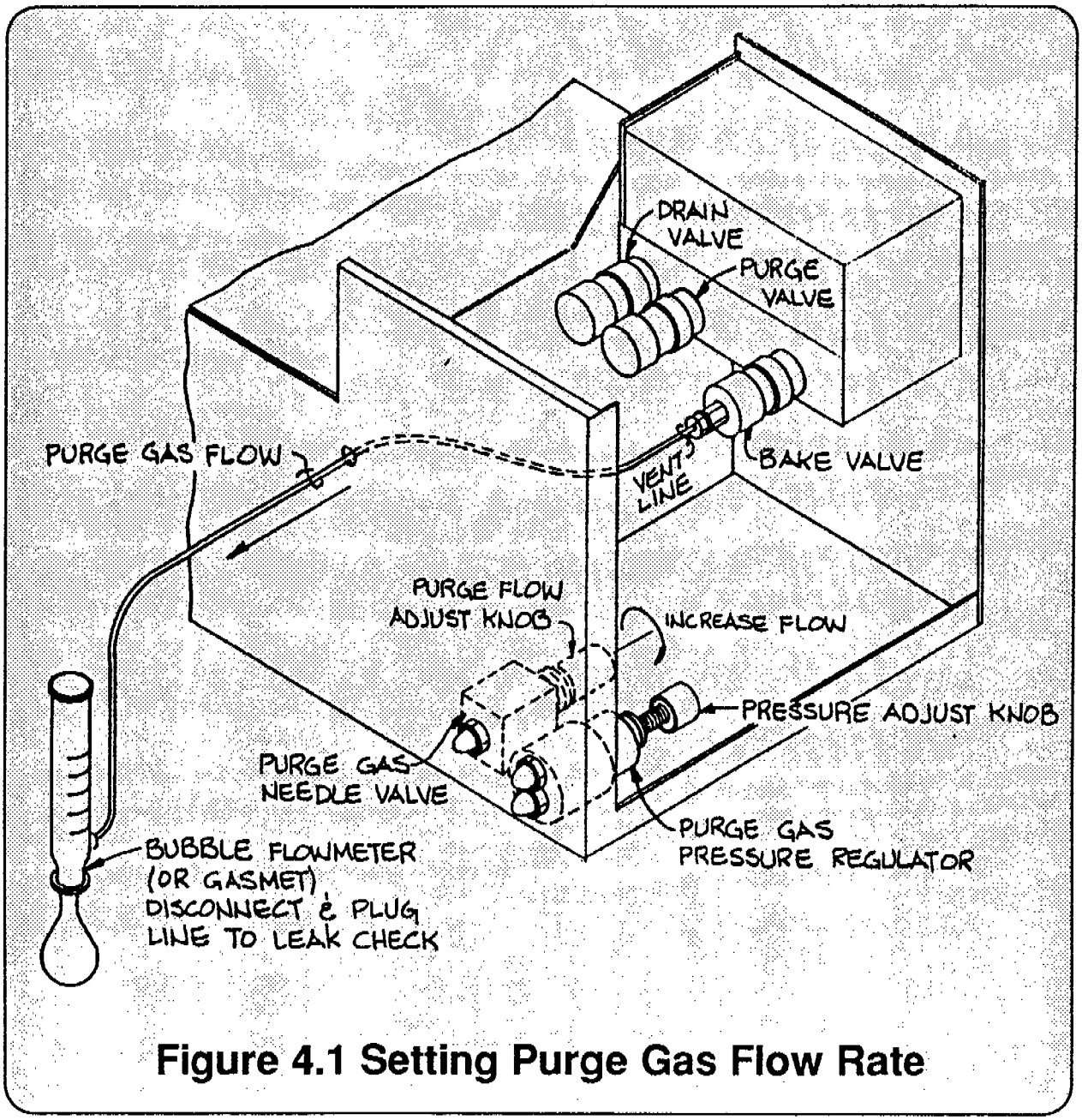


Figure 4.1 Setting Purge Gas Flow Rate

- Connect bubble flowmeter (OIC Part #177683) or Gasmet flowmeter (OIC Part #177053) to the vent port of the bake valve.

- Measure the flow rate of the purge gas and adjust purge gas flow controller as necessary to achieve the desired flow rate.

- Confirm that the purge gas pressure falls into the 5 to 7 psi range.

Purge Gas Leak Check

Confirm leak-tightness of the purge gas flow system by the following steps:

- While the system is in the PURGE state, inject a 5 ml sample of clean water, using the sample syringe (instructions for injecting a syringe sample are given later in this chapter under **Running a Sample**), and confirm visible purging.

- Close purge flow from bake valve by plugging the end of the vent line.

- Confirm that the purge gas pressure rises to 20 psi and that visible purging eventually stops. Rate will decay over a period of minutes. Check seals with electronic gas-leak detector if necessary to find leaks.

- Release flow restriction to regain purge flow. Confirm return of purge gas pressure to 5-7 psi.

Drain Check

Drain time can be checked with the sample valve in the Fill position if a drain line restriction is suspected. In this case, be prepared for the sample to be ejected from the syringe port onto the lab bench.

Check for the proper draining of a 5 ml sample from the purge vessel by the following:

- Confirm purge flow rate and leak-tightness per above, leaving sample in purge vessel.

- Turn on the 2nd FUNCT switch and press the DRAIN key.

- Time how long it takes to drain the 5 ml sample. About 8-10 seconds is normal. If drain time is significantly slower than this, inadequate gas flow to the purge tube, improper sample valve handle position, or a restriction in the drain line is probable.

Carrier Gas Leak Check

- Disconnect transfer line from GC injection port.

- Cover end of transfer line with septum to completely stop flow.

- Observe that the GC flow controller pressure (if gauge is available on GC) climbs rapidly to full scale or to regulator delivery pressure.

- Correct leak problems as necessary to achieve proper flow at end of transfer line.

Trap Bake

- Turn on the 2nd FUNCT switch and press the DRAIN key.

- Confirm that the trap temperature rises rapidly to the Bake Temp setpoint by watching the Temperature display and the trap status LEDs. Any sample which is in the purge vessel will automatically drain, then the unit will cycle to BAKE.

- The system will bake the trap until BAKE times out, or the timer may be turned off for extended baking.

- After trap bake is complete, advance the system to STANDBY using the RUN/STOP-STEP key. If the Bake timer is set to run, the system will time out of BAKE and return to STANDBY automatically.

Autosampling System Functional Checks

- Test the four key functions of the LSM:

  - PUMP: press to make pump run, press again to turn pump off.
  - WASH: press to make ASM needle arm swing to wash station.
  - LOWER: press to make ASM needle arm lower.
  - RAISE: press once to make ASM needle arm raise, press again to make arm swing to sample vial position.

- Install a 40 ml vial of water into the ASM rack.

- Set the 4460A DIP Switch #1 to ON (up).

- Press 2nd FUNCT CLEAR to reset the 4460A, then press the AUTO-ON/OFF and RUN/STOP keys to start the sampling sequence. The needle should lower into the vial, piercing the septum, and water should begin to fill the 5 ml sample loop.

- Note the fill rate of the sample loop by watching the 4460A Time display and the LSM drain line. The loop should completely fill with water within 15-17 seconds. Confirm the lack of bubbles in the draining water.

- After the Loop Fill Time has elapsed, the water in the loop will be injected into the purge vessel. Note the injection rate of the sample by watching the 4460A time display and the purge vessel. If the injection is not completed in 20 seconds (as indicated by a burst of coarse gas bubbles through the sample needle), a restriction or leak in the plumbing between the loop and the 4460A sample valve is likely. Another possibility is inadequate gas pressure to the loop due to an upstream leak. During this time, inspect for water and gas leaks throughout the LSM.

- After any necessary corrections have been made, advance to DESORB and let the sequence timer resume. Sample in the purge vessel should drain within 10 seconds.

- After DESORB, the ASM needle will raise from the vial, move to the rinse station, and the pump will begin pumping rinse water. Note the rinse rate by timing how long it takes to fill the loop with rinse water. The loop should completely fill in 30 seconds.
- This rinse should then be injected into the purge vessel. Watch for proper injection.
- A second rinse will then be pumped while the first rinse drains. Watch for proper operation.
- The second rinse will then be injected, then drained. Watch for proper operation.
- The sequence should then advance to BAKE, the needle should rise, and the tray should advance.
- After BAKE is complete, the needle should swing back over the next vial position.
- The 4460A autosampling system is now ready for operation. The ASM may be placed on top of the LSM.

Running A Sample

The preceeding functional checks should be completed before attempting to analyze a water sample. It is also assumed here that the GC column and detector have been properly interfaced, conditioned, and are ready for sample introduction. 4460A operating procedures are as follows:

Syringe Sample Loading

- Allow sample to come to ambient temperature prior to introducing it to the syringe.
- Remove the plunger from the sample syringe and attach a closed syringe valve.
- Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing.
- Replace the syringe plunger and compress the sample.
- Open the syringe valve and vent any residual air while adjusting the sample volume to the volume desired.
- Add any applicable surrogate spiking solution and internal standard spiking solution through the valve bore, then close the valve.

Because this process of taking an aliquot destroys the integrity of the sample for future analysis, you should fill a second syringe immediately after the first if you might have a need for a replicate.

Syringe Sample Injection

For the syringe injection sequence, DIP Switch #1 should be OFF and #6 should be ON.

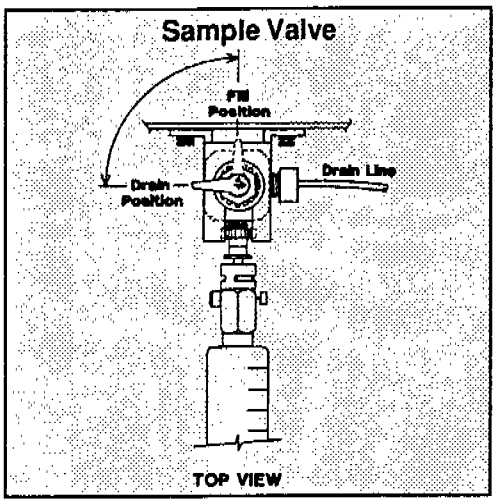
- When the system is in PURGE READY, press the AUTO-ON/OFF key if necessary, to place the system in AUTO ON (LED on).

- Press the RUN/STOP-STEP key to start the analysis sequence.

- Attach the syringe with valve to the 4460A sample valve syringe port.

CAUTION:
The sample may be injected in PURGE READY but without purge gas flowing it is possible to push liquid sample down through the frit.

- Open the syringe valve, then turn the Sample Valve handle to the fill position.



- Inject the sample into the purge vessel and close both valves. The sample will begin purging for the time set by the Purge Time setpoint. The system will then cycle through the states according to the setpoint values.

This wash can also be accomplished after the system returns to STANDBY or PURGE READY.

- After the sample cycles to BAKE, rinse the purge vessel by injecting a 5 ml flush of reagent water, turning the sample valve back to drain, allowing the rinse water to drain, and repeating with a second flush. If you wish to have the rinse drain rapidly, use the 2nd FUNCT DRAIN key (see next section).

- After the BAKE time has been completed, the system will cycle to STANDBY until the trap cools, then will wait in PURGE READY for the next sample.

Use of Manual DRAIN State

Anytime during the sequence the present sample may be drained by pressing the 2ND FUNCT DRAIN key. The sample will be drained as in DESORB W/DRAIN except that the trap is not placed in line with the GC column. The timer will restart to take the system through its pre-set drain time, then into the timed BAKE. The timer can be stopped at any time.

Use of CLEAR Command

Anytime during the sequence the timer, sample number, and replicate number can be re-zeroed, and the system returned to STANDBY, by pressing the 2nd FUNCT CLEAR key. Any sample in the system will not be affected. This command is particularly useful if the system has moved into the PURGE state during autocycling, but needs to be reset for manual sample injection.

Use of DESORB W/O DRAIN State

This state is inactive when the autosampler is enabled (DIP switch #1 ON) to prevent filling the purge vessel with sample after sample with no drain.

When the auto-drain switch is turned OFF, the DESORB state will not drain the sample in the purge vessel. In this case, the state status LED corresponding to DESORB will blink during DESORB, indicating the DESORB W/O DRAIN state. After DESORB W/O DRAIN has timed out, the system will cycle to BAKE as usual, then advance to STANDBY. The sample in the purge vessel can then be re-purged for purge efficiency experiments. If DIP switch #6 is turned ON, the sample in the purge vessel will be re-analyzed again and again until the pre-set sample number has been reached.

Autosampler Operation

Autosampler system operation with the 4460A is a very simple process, as seen by the outline of steps below. Skill required to keep the system operating properly is developed from an understanding of the operating principle and of the effects of changing the various time functions involved. Refer to **Overview of Method, Autosampler Injection** earlier in this chapter for a discussion of the operating principle.

Setting Autosampler Time Functions

All time functions pertaining to autosampler states are set to default values upon power-up. These values may be changed as necessary for particular applications. Each time function and its default setting is listed below:

State	Default Setting (sec)
LOOP FILL	30
LOOP INJECT	30
LOOP RINSE	FILL + 20
DRAIN RINSE	INJ + 15

These settings are appropriate for 5 ml samples. When other sample loop volumes are used, time settings for LOOP FILL and LOOP INJECT will need to be changed in accordance with the time necessary for proper sample loop filling and transfer to the purge vessel. Times of LOOP RINSE and DRAIN RINSE are automatically reset when FILL and INJECT times are changed.

Instructions for Operation

- Turn DIP Switch #1 ON to enable the autosampler sequence.
- Turn DIP Switch #6 OFF to allow automatic cycling past PURGE READY.
- Place 40 ml sample vials into the vial racks of the ASM and check the wash vessel reservoir for sufficient rinse water.
- Set the number of samples to be run and the number of replicates to be analyzed from each vial.
- Press 2nd FUNCT CLEAR to return the system to STANDBY and to reset the sample and replicate counters.
- Press the AUTO-ON/OFF key to place the system in AUTO ON (LED on).
- Press the RUN/STOP-STEP key to start the automatic cycle.

Standards Injection Module Operation

Principle of Operation

The internal bore of a 4-way valve is used to measure and dispense a precise 10-microliter volume of standard into the fluid flow stream. The standards solution is loaded into a motor-controlled 10 ml syringe connected to this valve. The water

sample to be purged (5-25 ml) passes from the autosampler through the valve on its way to the purge vessel of the sample concentrator. As it does so, it sweeps the contents of the valve into the purge vessel. An electric actuator is used to rotate the valve either in line with the syringe to fill its internal bore with standard solution, or in line with the sample stream to dispense its contents into the purge vessel.

Pre-Operational Steps

- Set the ON/BYPASS switch to BYPASS.
- Retract the standards syringe plunger to remove the syringe for filling. Alternatively, advance the syringe to clear it of old standard, then place the waste line in a vessel of new solution and retract the syringe to fill it.
- If the syringe was removed to fill it with new standards solution, insure that bubbles have been removed and remount the syringe, then advance the plunger arm to engage the barrel.
- Advance the plunger to flush about a milliliter of new solution through the injection valve.
- Confirm the number of injections is set to deliver the needed volume of solution.
- Set the ON/BYPASS switch to ON to enable automatic operation. This step will inject the 10 µl volume presently in the valve bore, so the 4460A should be placed in BAKE or DRAIN to rid the system of this initial aliquot.

Running an Analysis

Once the SIM is enabled per the above section, no further attention is needed except to inspect the syringe at regular intervals to monitor its usage of solution. The solution is typically replenished every one to three days, depending on the volume injected with each sample.

Needle Sparging

The instructions given here in the manual are to be used only as guidelines and that actual methodology may require different parameters (weights, volume, etc.) than stated.

Purge and trap analysis using a needle sparger is very similar in operation to analysis using a fritted sparger. There are two main differences however. First is the purging efficiency of a needle sparging apparatus which in general is lower than that for a fritted (or dispersed bubble) sparge. This is often overcome by either increasing the purge time, elevating the temperature of the sample during sparging, or a combination of both.

The other difference is that there is not an automatic draining of the sample at the end of the analysis. Therefore the purge vessel must be manually drained at the end of each cycle. This is accomplished by removing the vessel and emptying the spent sample, then either cleaning the vessel for a new sample or installing a fresh purge vessel. Alternatively, liquid samples may be drawn backout into a syringe through the needle.

The following two sections describe the analysis of liquid samples and solid samples respectively when using a needle sparger on the 4460A.

Instructions for Needle Sparging Liquid Samples

When analyzing samples with a needle sparger, DIP switch #1 should be OFF and #6 should be ON.

- Follow the instructions outlined earlier for the proper syringe sample loading technique.
- If heated sample sparging is to be performed, confirm that the heater jacket is in place and that the correct EXT TEMP is set.
- When the system is in PURGE READY, press the AUTO-ON/OFF key if necessary to place the system in AUTO ON (LED on).
- Attach the syringe with valve to the 4460A sample valve syringe port.
- Open the syringe valve, then turn the sample valve handle to the fill position.
- Inject the sample into the purge vessel and close both valves.
- Press the RUN/STOP-STEP key to start the analysis sequence. The sample will begin purging for the time set by the purge time setpoint and continue through the other states according to the setpoint values.
- After the 4460A cycles to BAKE, loosen the lower 1/2 inch knurled nut and slide the purge vessel off.
- Clean and rinse the vessel before reinstalling or install a cleaned and baked purge vessel and tighten the lower nut back fingertight.
- When the 4460A cycles to PURGE READY, proceed with the next sample as above.

Instructions for Needle Sparging Solid or Sludge Samples

When analyzing samples with a needle sparger, DIP switch #1 should be OFF and #6 should be ON.

- Tare a clean, dry sparge vessel on an analytical balance. Write down this mass.
- According to the method of analysis being followed, weigh a known amount of the sample into the purge vessel. A 1-5 gram sample is typical depending on expected analyte concentration.
- Weigh the purge vessel with the sample again. Write this mass down and calculate the mass of the sample.
- Install the vessel with sample on the 4460A with the neck of the purge vessel inserted properly into the knurled nut and reducing union. Tighten the knurled nut fingertight for a leak-free seal.
- If a heated sparge is to be performed, slip on heater jacket and confirm EXT TEMP is set to correct temperature.
- Using a reagent water free of volatile organic compounds fill a 5 ml (or 25 ml depending on method) syringe as described earlier in this chapter (see **Syringe Sampling Loading**).

The sequence of steps from weighing the sample into the vessel to attaching vessel to the 4460A must be carried out quickly and performed in an environment free of ambient volatile compounds.

- When the system is in PURGE READY, press the AUTO-ON/OFF key if necessary, to place the system in AUTO-ON (LED on).

- Attach the syringe with valve to the 4460A sample syringe port.

- Open the syringe valve, then turn the sample valve handle to the fill position.

- Inject the reagent water into the purge vessel and close both valves.

- Press the RUN/STOP-STEP key to start the analysis sequence. The sample will begin purging for the time set by the purge time setpoint and continue through the other states according to the setpoint values.

- After the 4460A cycles to BAKE, loosen the lower 1/2 inch knurled nut and slide the purge vessel off.

- Clean, rinse and dry the vessel before weighing the next sample or perform the subsequence analysis with a cleanded and baked vessel.

- When the 4460A cycles to PURGE READY, proceed with the next sample as above.

Chapter 5
Maintenance and Troubleshooting

In the last chapter, a summary of the 4460A method of operation, a set of pre-operational checks, and a step-by-step guide for running a sample are given. The pre-operational checks are used there to confirm proper function before operation. They can also be used for diagnostics. If a symptom develops in operation of the 4460A or the autosampler, the functional checks can be used to isolate the problem. In this case, read these discussions of the use of the diagnostic tools at your disposal, then follow the troubleshooting guide.

Trap Maintenance

The only component of the 4460A which requires periodic maintenance is the trap. After a period of extended use, a trap may begin to degrade, causing loss of performance. Symptoms include a loss of sensitivity of selected components in a standard run and increasing pressure required to maintain 40 ml/min flow during PURGE. When a trap is suspected of failing, it should be replaced. We recommend that each new trap be conditioned overnight at 200°C with helium flow before use. To condition a new trap, simply place the system in BAKE without the timer running and set the Bake Temp to 200°C. We offer a Trap Conditioning Module so that a new trap can be pre-conditioned without tying up the 4460A. It is also good practice to keep a "reference" trap sealed and on hand, in case you want to test system performance using a trap you know to be good.

4460A Diagnostic Tools

You have two powerful diagnostic tools built into the 4460A. These are the manual actuator button for the trap valve and the purge flow controller/pressure gauge. If your GC has a pressure gauge tied to the output of its carrier gas flow controller to measure column head pressure, it can be a third diagnostics tool.

Troubleshooting the 4460A has been condensed to a set of step-by-step procedures outlined in **Chapter 4** under **4460A Functional Checks**. In order for you to fully understand the purpose of these checks, the use of the three diagnostic tools is explained here.

Manual Actuator Button

This button on the trap valve actuator solenoid allows you to rotate the trap valve without the use of the 4460A sequence. It will even rotate the valve with the power off. The manual activator allows you to confirm that the trap valve is able to fully rotate. Inability to rotate can be caused by insufficient actuator gas pressure or a frozen actuator or valve. If the trap valve does not rotate at all, the components on the trap will not be desorbed onto the GC column but will be desorbed out to vent, causing a complete loss of response. Incomplete valve rotation is caused by misalignment of the trap valve and its actuator, and will cause an increased or complete restriction to gas flow through the valve. Restrictions of this nature can be detected

using the second diagnostic tool, as discussed next.

Purge Flow Controller/Pressure Gauge

The 4460A uses a flow controller rather than a simple needle valve for purge flow control. This design allows an exact flow rate to be maintained regardless of variations in trap restriction. The pressure gauge is connected to the output of the flow controller so that the gas pressure required to maintain the set flow can be monitored. In PURGE, the purge gas flows sequentially through:

- the rear hydrocarbon scrubber
- the purge valve
- the purge vessel
- the trap
- the bake valve to vent

The total restriction caused by these components generates a backpressure of about 5-7 psi in order to maintain 40 ml/min purge flow. If a pressure above about 7 psi is observed during PURGE, one of these five components is causing too great a restriction. Typically, 3 psi of the pressure drop occurs across the hydrocarbon scrubber and another 3 psi occurs across the trap. The remaining components do not cause a measureable drop unless malfunctioning. If too great a pressure is observed, find the restriction by pressure-isolating the first component, then the first two together, then the first three, etc.

If too *low* a pressure is noted during PURGE, a leak involving one of these components is likely. The trap connections to their bulkhead fittings is the best place to look first. To aid in locating a leak of this type, plug the purge vent coming out of the bake valve (this is the same port that is used to measure the purge gas flow rate). When it is plugged, purge flow will cease so the flow controller will increase purge pressure in an attempt to maintain the purge flow rate. This rise in pressure is shown on the pressure gauge, which should stabilize at 20 psi. At this point, the components listed above will all be pressurized to 20 psi and finding a leak is made easier.

The pressure gauge readings in other states are also useful to know. Approximate values for each state are:

· STANDBY/PURGE READY	20 psi
· PURGE →	5-7 psi
· DRY PURGE	5-7 psi
· PURGE COMPLETE/DESORB READY	20 psi
· DESORB W/DRAIN	2-3 psi
· BACKFLUSH BAKE	2-3 psi →
· DRAIN	2-3 psi
· DESORB W/O DRAIN	2-3 psi

Those states in which the gauge reads 20 psi are ones during which purge gas is not flowing. When the purge valve is shut off during these states, the flow controller opens up to its supply pressure in an attempt to supply its pre-set flow. This supply pressure is called the system pressure and is regulated to 20 psi by the internal pressure regulator. This pressure also drives the bake flow and the fill and inject gas flows of the LSM.

Those states in which the gauge reads 2-3 psi are ones during which the trap is not in line with the purge gas, so less pressure is required to maintain the pre-set flow. If the pressure during these states rises to an abnormally high level, then a blockage, crimp, or other restriction has developed between the purge vessel and its draining

mechanism. In this case, check the sample needle, the sample valve, the drain valve, and the drain line to waste.

Carrier Flow Controller/Pressure Gauge

Built into your GC is a flow controller for regulating the flow of carrier gas through the column. If this carrier gas flow controller has a pressure gauge connected to its outlet to measure column head pressure, then it can be used to isolate any loss of response or change in retention times. The carrier gas flow rate for packed column purge-and-trap analysis is typically 30-40 ml/min, which requires 30-50 psi at the head of the column to produce. The carrier flow rate for 0.53 mm ID capillary column purge-and-trap analysis is typically 7-10 ml/min, which requires only 5-10 psi to maintain. As the column temperature increases, more pressure is required to maintain the set flow rate. When the 4460A has been connected to the GC, the flow from the controller goes through any carrier scrubber, then through the 4460A trap valve, back out the heated transfer line, and into the GC injector and column. Once the system is properly connected together, make note of the carrier pressure required to maintain the desired column flow rate at the initial column temperature. Also note the increase in pressure with the temperature ramp to be used for analysis. If you later observe a higher pressure than normal, a restriction in the trap valve or the injector is likely. If you notice a lower column pressure than normal, a leak in the components between the flow controller and the column has developed. In this case, the injector septum on the GC is the most suspect. If you notice a shift in retention times of each of the components, examine the carrier pressure gauge for abnormal pressure. In this case, if nothing is wrong with a capillary carrier pressure, the GC flow controller may be unsuitable for flow rates less than 10-20 ml/min.

Troubleshooting

4460A

To troubleshoot the 4460A, first turn off the autosampler DIP switch (#1) and disconnect the gas lines between the 4460A and the LSM, if you have the autosampling system connected. Next, review the use of the diagnostic tools in the above section. Then, depending on the symptom, either change the trap or perform the **4460A Functional Checks** in **Chapter 4.** These are:

- Pre-Power-Up Checks
- Confirming Trap Valve Rotation
- Setting Purge Gas Flow Rate
- Purge Gas Leak Check
- Drain Check
- Carrier Gas Leak Check
- Trap Bake

If the 4460A fails to pass a check, diagnose and correct the problem based on the above discussions. If the system passes these checks, the fault is not likely to be with the 4460A.

Autosampling System

To troubleshoot the autosampling system, connect the gas lines between the 4460A and the LSM and turn on the autosampler DIP switch (#1). Next, perform the **Autosampling System Functional Checks** in **Chapter 4.** Correct any leaks as necessary to successfully complete the check list.

PART NAME	PART #	U/M	XPND
Ferrules			
BR 1/16 Tube Back	176314	ea	*
BR 1/16 Tube Front	177156	ea	*
BR 1/8 Tube Set	128082	ea	*
PTFE 1/16 Tube	177626	ea	*
SS 1/16 Tube	112433	ea	*
TFE 1/16 Ring	175952	ea	*
TFE 1/4 Tube	175978	ea	*
VSP 1/8 - 1/16 Tube	177252	ea	*
Fittings			
Adaptor - BR HP x 1/8 Tube	184754	ea	
Adaptor - BR 1/16 Tube x 10-32	179010	ea	
Adaptor - BR 10-32 x 1/16 Hose	166191	ea	
Adaptor - Kel-F F-Luer 1/4-28	169468	ea	*
Adaptor - PP 10-32 x 1/16 Hose	179051	ea	*
Bushing - Kel-F 1/4-28	169450	ea	*
Coupling - BR 10-32	176033	ea	
Injection Port - Kel-F 26 Ga 2 in.	175902	ea	
Nipple - SS 10-32 O-Ring	176041	ea	
Nut - NI/BR 10-32 x 5/16 Hex	179531	ea	
Nut - BR/NI 1/16 Tube Male	176306	ea	
Nut - BR 1/8 Tube Female	128108	ea	
Nut - SS 1/16 Tube Female	176017	ea	
Nut - SS 1/16 Tube Male	169640	ea	
Plug - BR 10-32	167652	ea	
Tee - BR Swivel 10-32	179309	ea	
Tee - SS/AU Purge Vessel	185074	ea	
Tee - SS 1/16 Tube M/Female	175770	ea	
Trap Bulkhead	185058	ea	
Union Blkhd - BR 1/8 Tube	124420	ea	
Union Blkhd - SS 1/16 Tube	175829	ea	
Interface Assemblies			
Carrier Antek 3K	178327	ea	
Carrier HP5880	178236	ea	
Carrier HP5890	178244	ea	
Carrier Shimadzu GC9A	178228	ea	
Carrier Std GC	178251	ea	
Carrier Varian 3400	179820	ea	
Low-Vol HNU	178699	ea	
Low-Vol HP5880	177824	ea	
Low-Vol HP5890	176900	ea	
Low-Vol Shimadzu GC9A	177741	ea	
Low-Vol STD GC	178533	ea	
Low-Vol Tracor 540	182337	ea	
Low-Vol Tracor 565	182345	ea	
Low-Vol Varian 1040	177766	ea	
Low-Vol Varian 1045	177758	ea	
Low-Vol Varian 3400	179838	ea	

PART NAME	PART #	U/M	XPND
Support Items			
Flowmeter - 10 ml Bubble	178459	ea	*
Flowmeter - Differential Gas	178053	ea	
Heater Assy - 25 ml Flexible	182162	ea	*
Heater Assy - 5 ml Flexible	182170	ea	*
Hook-Up Kit	177592	ea	
Manual - Operating Procedures	177354	ea	
Regulator - Compressed Air SS Diaphram	174714	ea	
Regulator - Helium SS Diaphram	174730	ea	
Regulator - Hydrogen SS Diaphram	174722	ea	
Startup Kit	177618	ea	
Syringe - 10 µl x 2 in. Needle	167545	ea	*
Syringe - 10 ml SIM	182006	ea	*
Syringe - 25 ml Sample	172461	ea	*
Syringe - 5 ml Sample	177659	ea	*
Syringe Kit	177634	ea	
Tubing - CU 1/8 x .060 ID	111427	ft	*
Valve - Sample Syringe	177642	ea	
Traps			
Blank	169509	ea	45 65
Tenax Only (#1)	169559	ea	*
OV-1/Tenax (#2)	169567	ea	*
OV-1/Tenax/Silica Gel (#3)	169517	ea	*
OV-1/Tenax/Silica Gel/Charcoal (#4)	169525	ea	*
Valves			
3 Port Sample 1/4-28	169484	ea	
3 Way Solenoid w/Viton	175671	ea	
6 Port Rotary SS w/Act	175225	ea	
Actuator Solenoid 4-Way	184614	ea	
Needle Sparging			
Ferrule - Kel-F 1/16 Fl-Fr	187203	ea	*
Ferrule - PTFE 1/16 Tube	177626	ea	*
Ferrule - TFE 1/16 Ring	175952	ea	*
Ferrule - TFE 1/4 Tube	175978	ea	*
Ferrule - TFE 1/2 (Front and Back)	187278	ea	*
Ftng - Nut Al Fl-Fr 1/4 x 28-1/16L	186783	ea	*
Ftng - Union Reducing 1/2 x 1/4 in. Au/SS	187245	ea	*
Ftng - Union SS 1/16 Tube Male w/Nuts	165747	ea	*
Needle - 5/25 ml Needle Sparging 16 Ga	185082	ea	*
Tube - 5 ml Needle Sparge	187484	ea	*
Tube - 25 ml Needle Sparge	188508	ea	*
Tube - SS/Au 1/4 in. x 2 mm ID	187526	ea	*
Tube - SS Needle Sparge Adaptor	189308	ea	*

Parts For Autosampling System

PART NAME	PART #	U/M	XPND
Cable Assy - 4460A/LSM Interface	175457	ea	
Cable Assy - LSM/ASM Interface	175473	ea	
Ftng - Tee PTFE 1.5 mm ID	170431	ea	*
Ftng - Union Kel-F 1/4-28	178186	ea	*
Module - Autosampling	168402	ea	
Module - Autosampling - Rebuilt (req. exchg)	182675	ea	
Module - Loop Sampling - Rebuilt (req. exchg)	182683	ea	
Module - Loop Sampling w/Kit	177057	ea	
Module - Loop Sampling w/o Kit	177049	ea	
Screw Cap - Open Hole for 40 ml	168147	ea	*
Screw Caps - Open Hole for 14 ml (50/pk)	174558	pk	*
Screw Caps - Open Hole for 40 ml (50/pk)	174532	pk	*
Septa - Teflon Faced for 14 ml (50 pk)	169054	pk	*
Septa - Teflon Faced for 40 ml (50/pk)	173211	pk	*
Tubing - TFE 1/16 x .010 ID	139065	ft	*
Tubing - TFE 1/16 x .031 ID	145591	ft	*
Tubing - TFE 1/8 x .063 ID	147901	ft	*
Vials - Autosampler 14 ml (344/bx)	169038	bx	*
Vials - Autosampler 40 ml (100/bx)	173196	bx	*

ASM Parts

Collar - Hold Down Foot, Septum Piercing	174029	ea	
Collet Assy	179283	ea	
Foot - Hold Down, Septum Piercing	174037	ea	
Needle - Septum Piercing 3.5 in. 15 Ga	173980	ea	*
Needle - Septum Piercing 7 in. 18 Ga	178723	ea	*
Power Cord - Jumper	176992	ea	
Rack - ASM 16 mm Tube 20/Set	173170	ea	
Rack - ASM 18 mm Tube 20/Set	174235	ea	
Rack - ASM 28 mm Tube 10/Set	173162	ea	
Rod - SS Hold Down Foot, Septum Piercing	174011	ea	
Rod - SS Pipet ASM	178368	ea	
Septum Piercing Kit	178046	ea	
Spacer - Wash Station	178707	ea	
Tube - SIL 3/16 ID, Sample Pump	177247	ea	*
Tubing - TYG 3/16 OD x 1/16 ID	174003	ft	*
Wash Vessel	178731	ea	*

LSM Parts

Control Board	176984	ea	
Ferrule - Kel F 1/8 Tube	18/6742	ea	*
Ftng - Adapt PP 1/4-28 x 1/4 Barb	170994	ea	*
Ftng - Coupling PP 1/4-28	166274	ea	*
Ftng - Elbow BR Swivel 10-32	179382	ea	
Ftng - Tube End PP 1/4-28 x 1/16 B	166331	ea	*
Ftng - Tube End PP 1/4-28 x 1/16 L	166282	ea	*
Ftng - Tube End PP 1/4-28 x 1/16 R	166307	ea	*
Ftng - Tube End PP 1/4-28 x 1/8 G	166357	ea	*

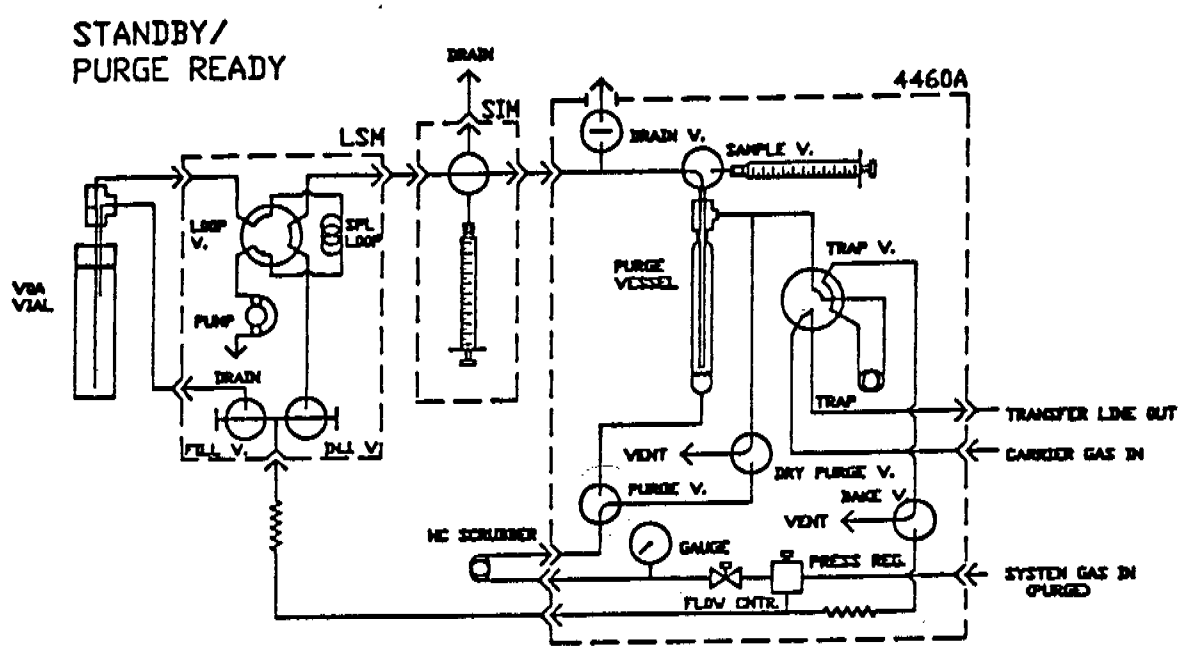
PART NAME	PART #	U/M	XPND
Ftng - Tube End PP 1/4-28 x 1/8 W	179424	ea	*
Ftng - Tube Nut AL Black	186734	ea	*
Ftng - Tube Nut AL Yellow	186726	ea	*
Fuse - 2 Amp	177453	ea	*
Pump - Sample, Peristaltic 18 ml/min	177239	ea	
Sampe Loop - 5 ml NI	186700	ea	
Sample Loop - 25 ml NI	186767	ea	
Sample Loop - 25 ml TFE	177221	ea	*
Sample Loop - 5 ml TFE	166505	ea	*
Startup Kit	177031	ea	
Valve - 3 Way Manifold w/Viton	179325	ea	
Valve - 6 Port TFE w/Actuator	161498	ea	

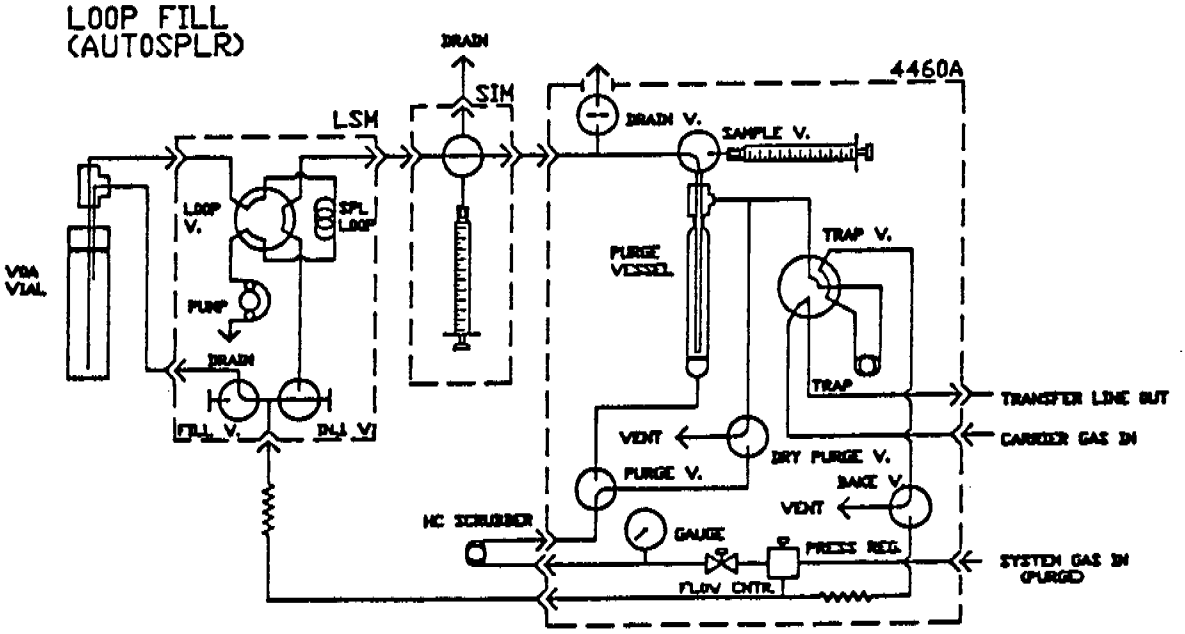
Chapter 7
Flow Schematics

Chapter 1, Operational Concepts discribes the various instrument states and **Chapter 4, Overview of Method** discussed what happens during the automatic sequence through these states. This chapter presents schematic flow diagrams for each of the states, showing positions of valves and directions of flow.

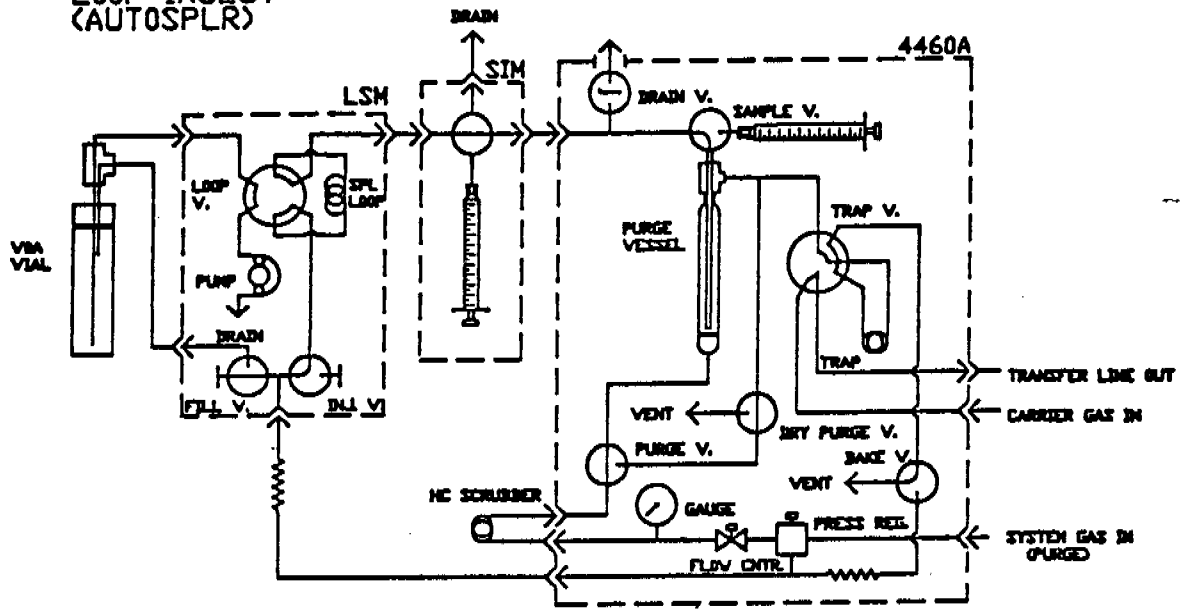
Flows using the VOA Vial Autosampler and Standards Injection Module are shown as well. Two sets of flow diagrams are presented: one for frit sparging and another for needle sparging. The states are placed in order of their normal sequence. Those states which are only active when the autosampler DIP switch (#1) is on are marked by "AUTOSPLR" in parentheses.

Frit Sparging

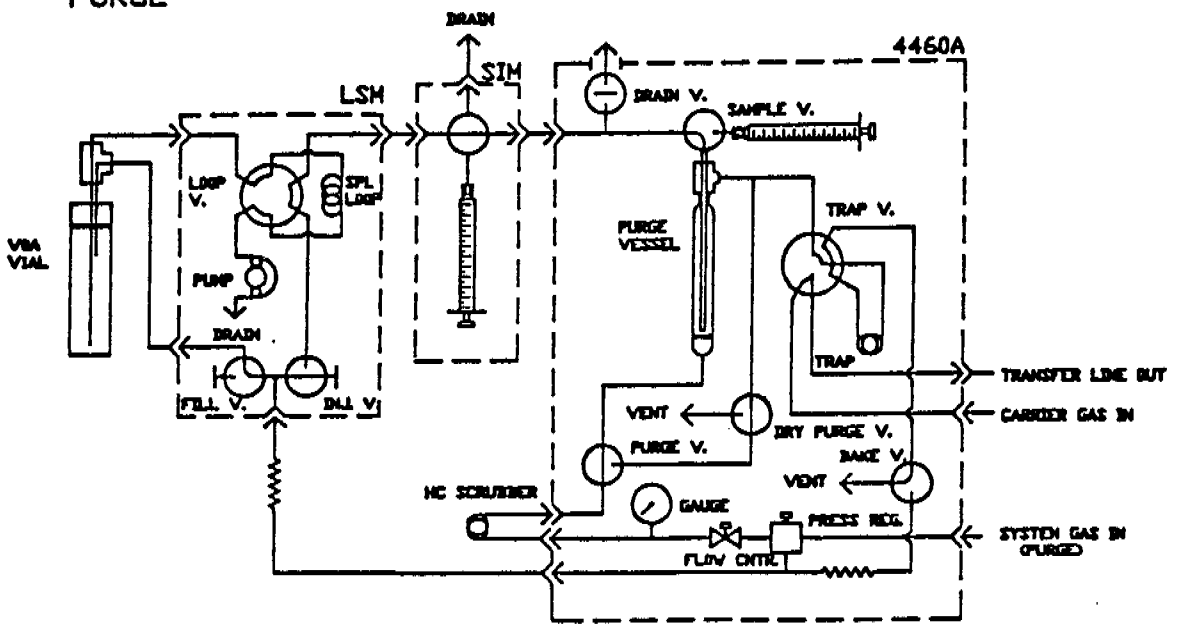




LOOP INJECT
(AUTOSPLR)

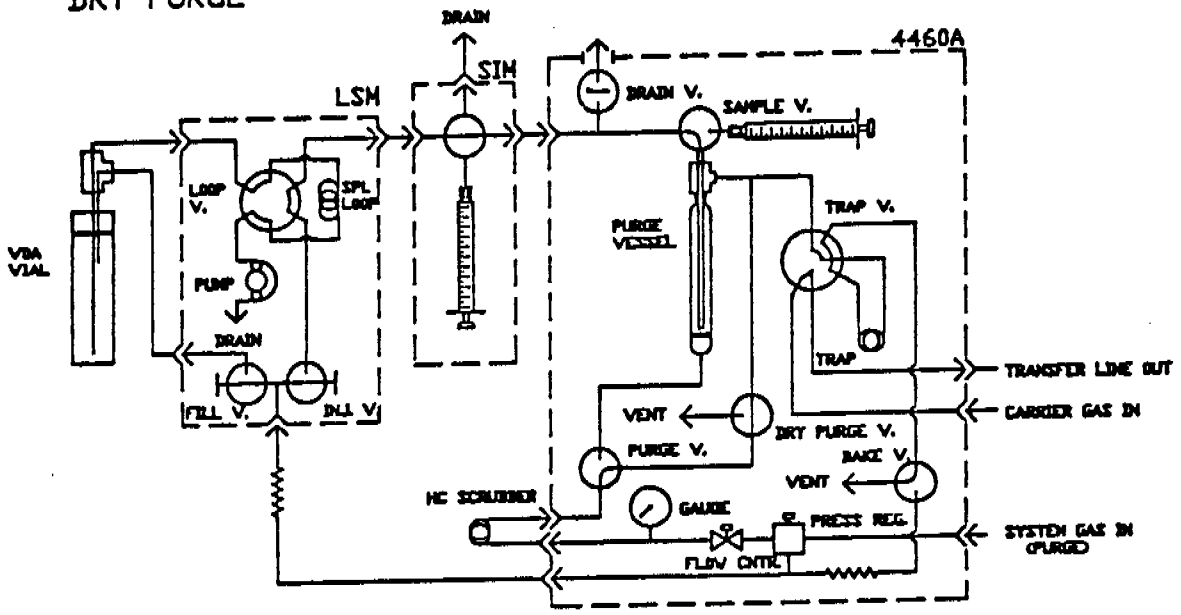


PURGE

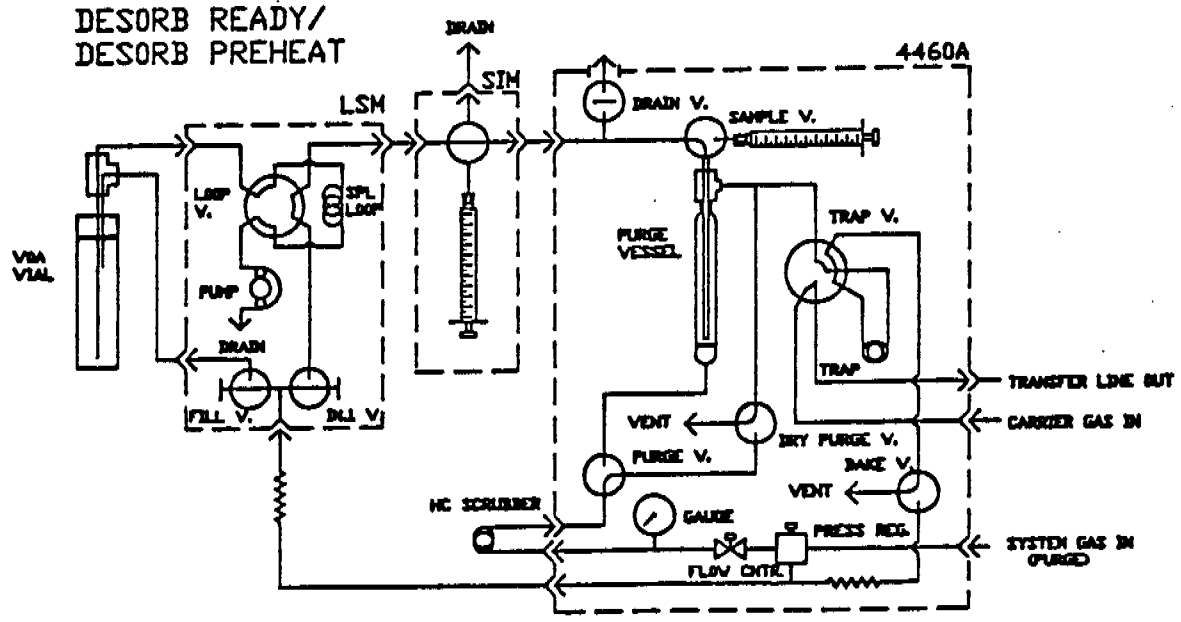


Frit Sparging

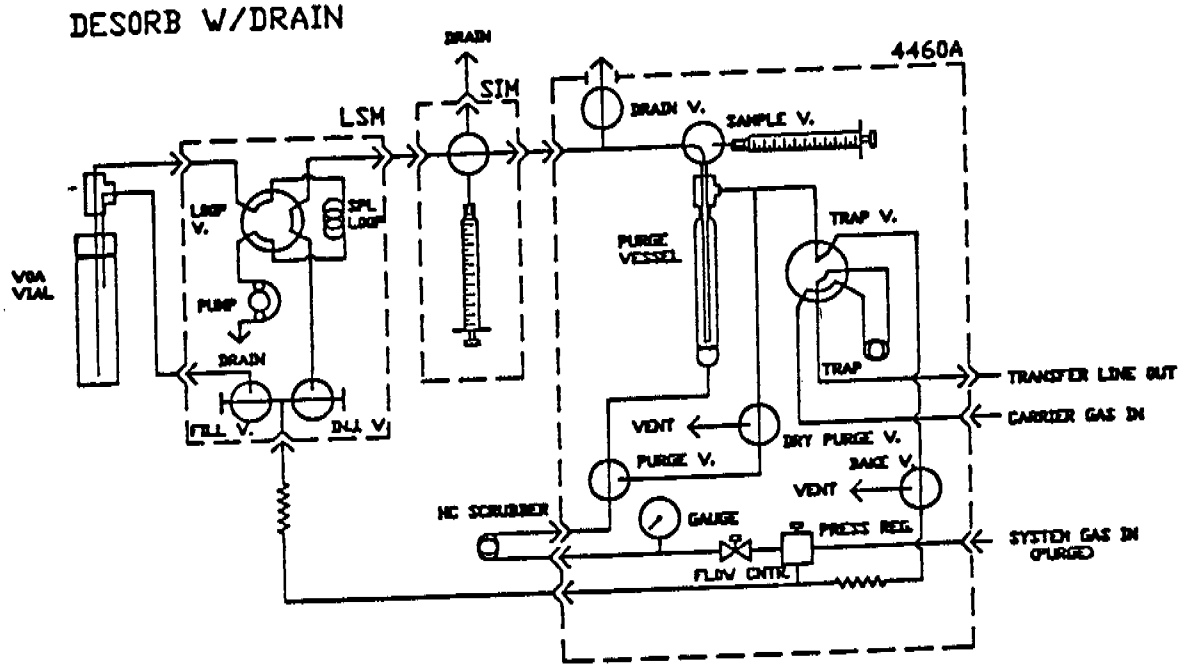
DRY PURGE



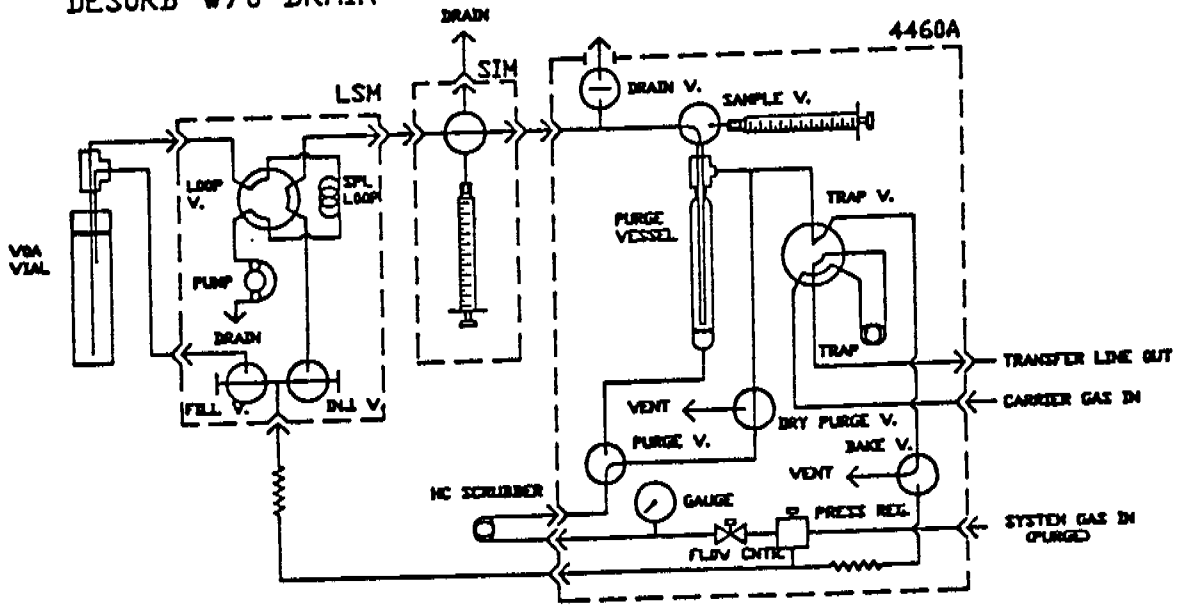
PURGE COMPLETE/
DESORB READY/
DESORB PREHEAT



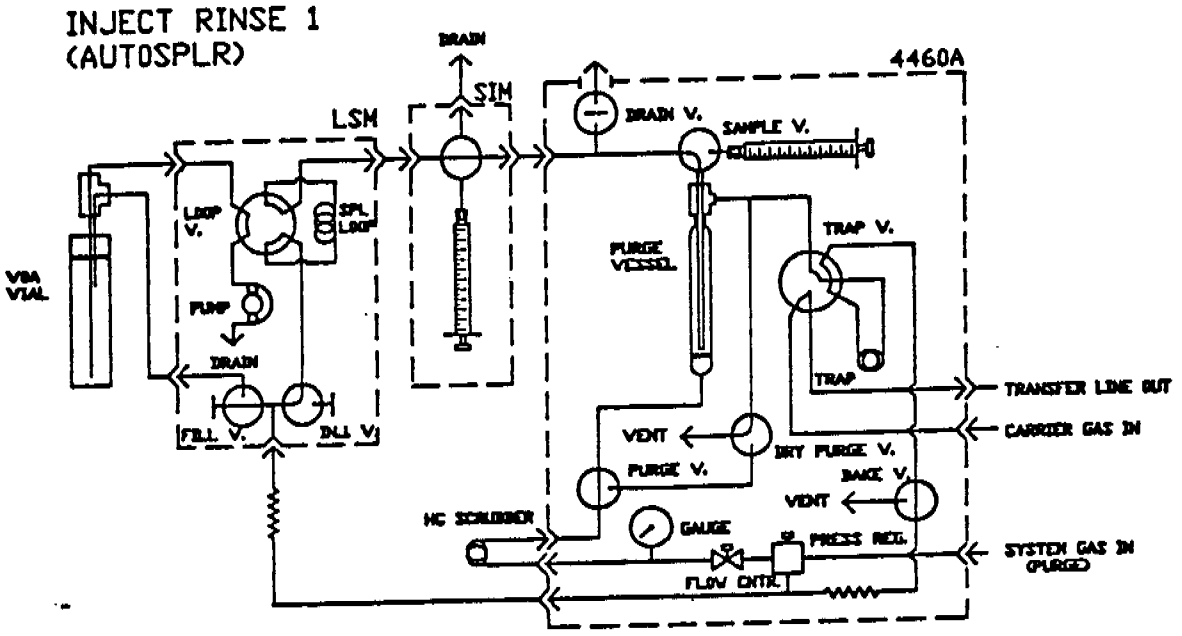
DESORB W/DRAIN

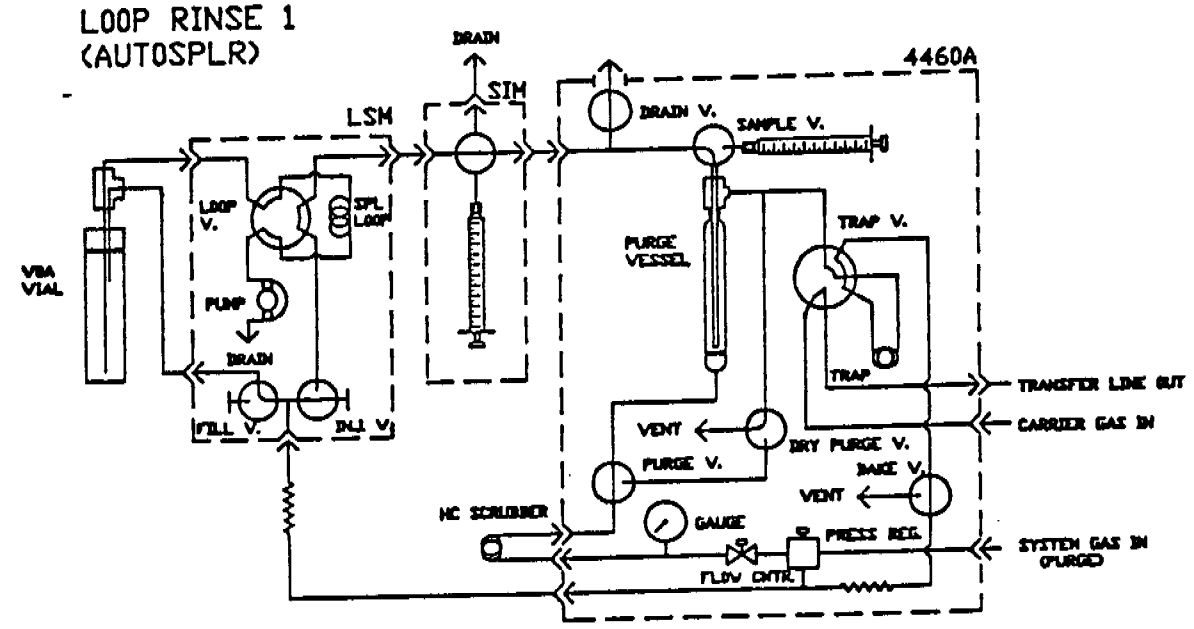


DESORB W/O DRAIN

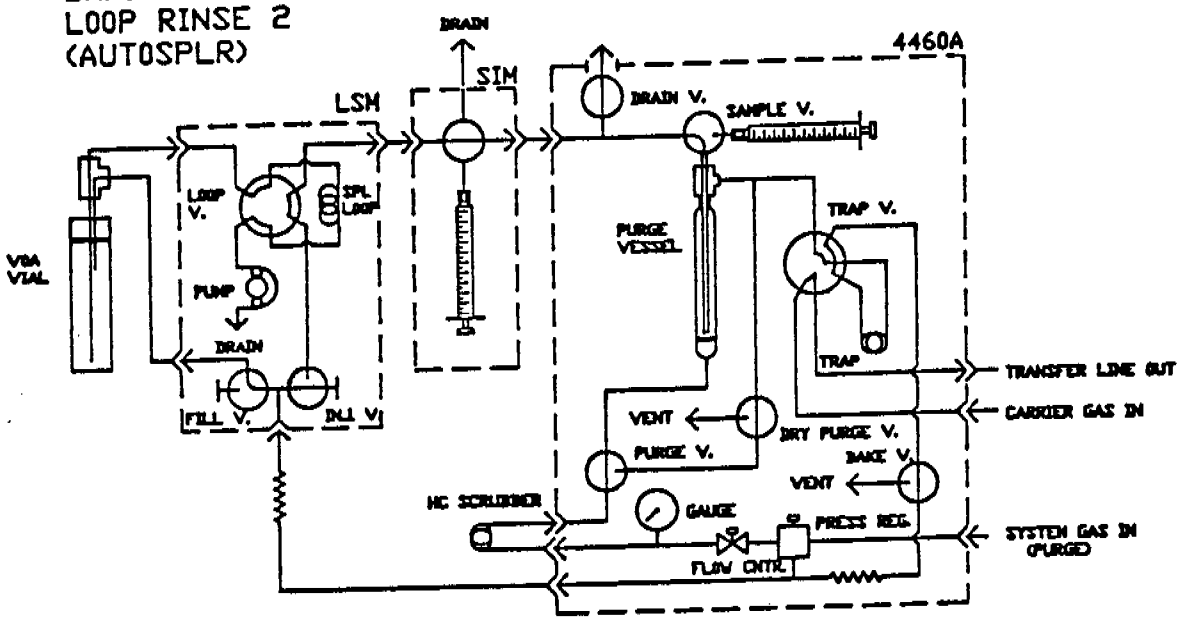


Frit Sparging

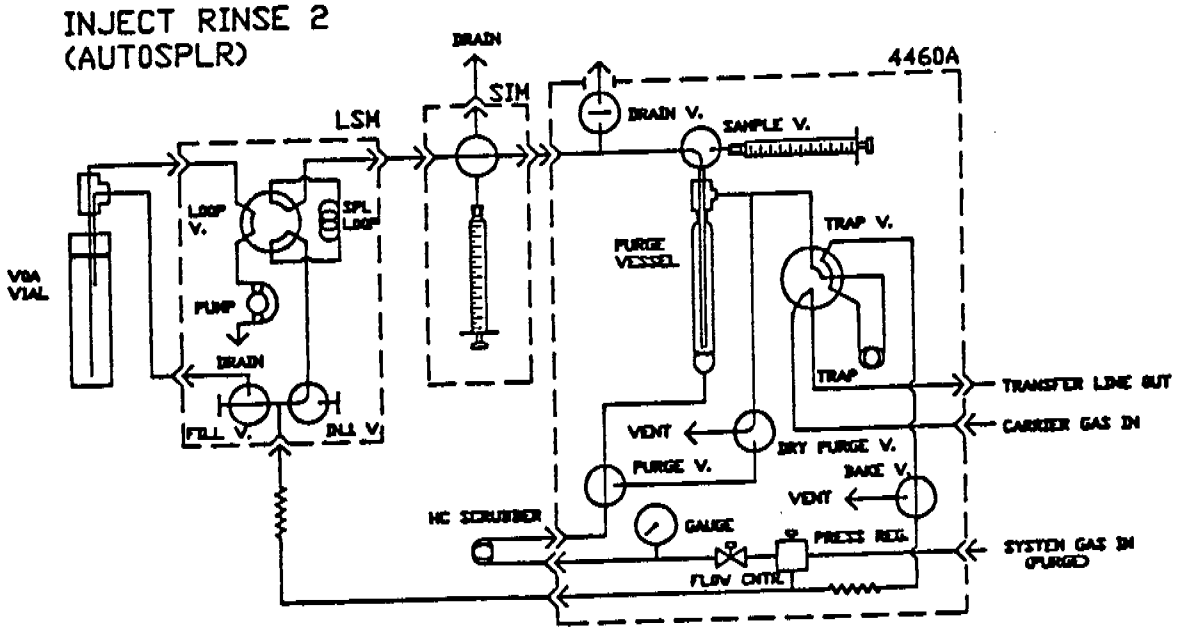




DRAIN RINSE 1/
LOOP RINSE 2
(AUTOSPLR)

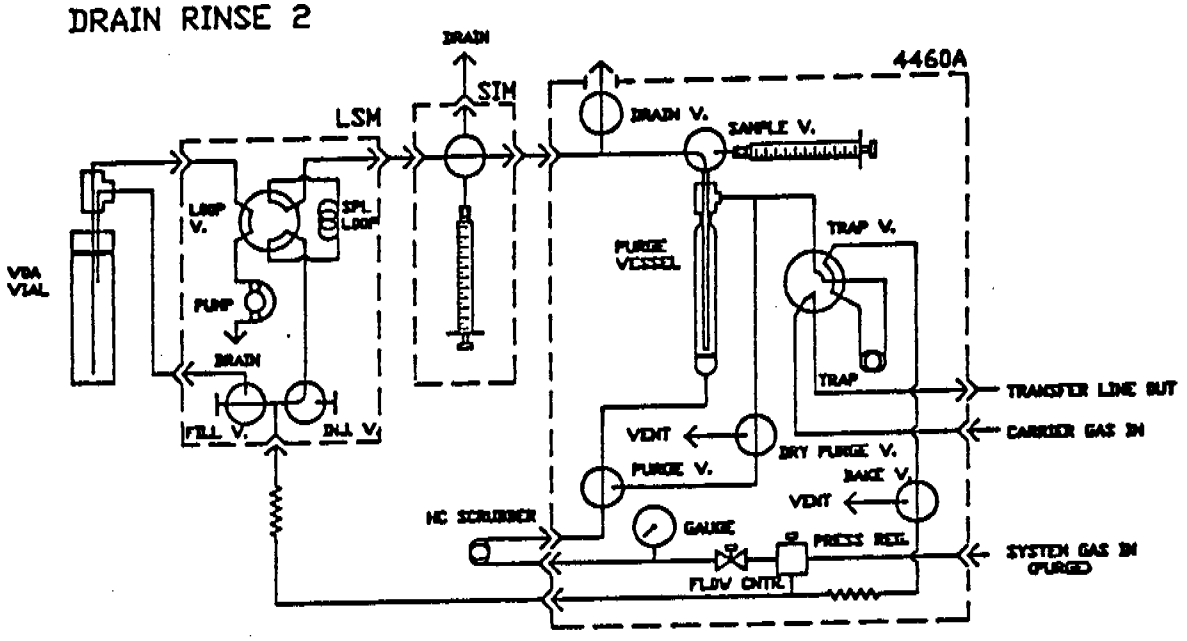


INJECT RINSE 2
(AUTOSPLR)

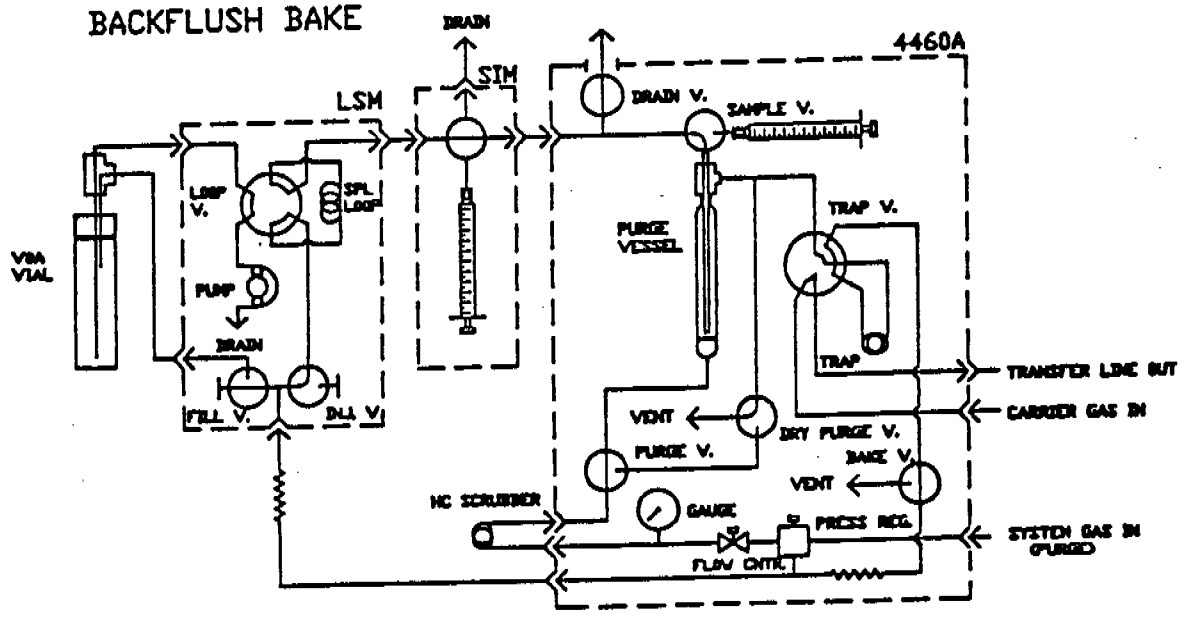


Frit Sparging

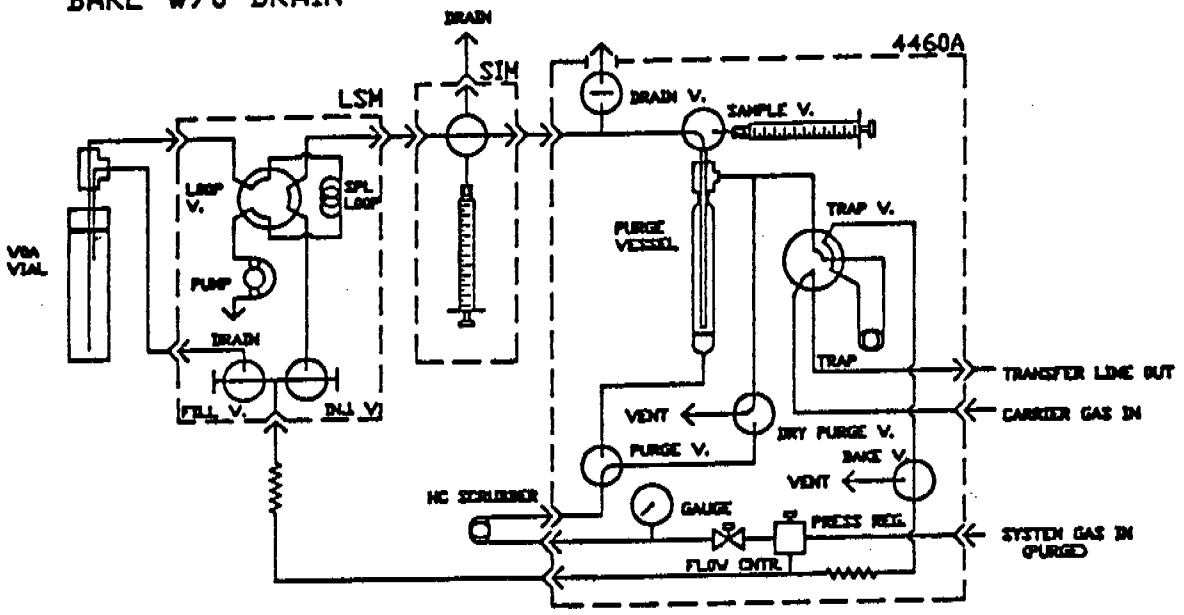
DRAIN RINSE 2



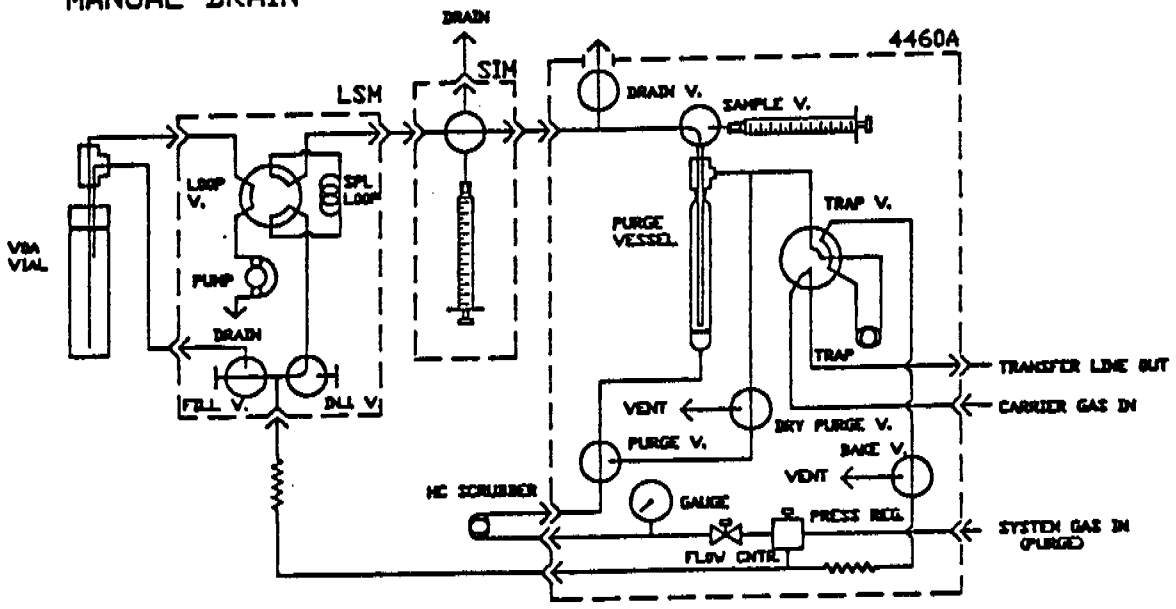
BACKFLUSH BAKE



BAKE W/O DRAIN



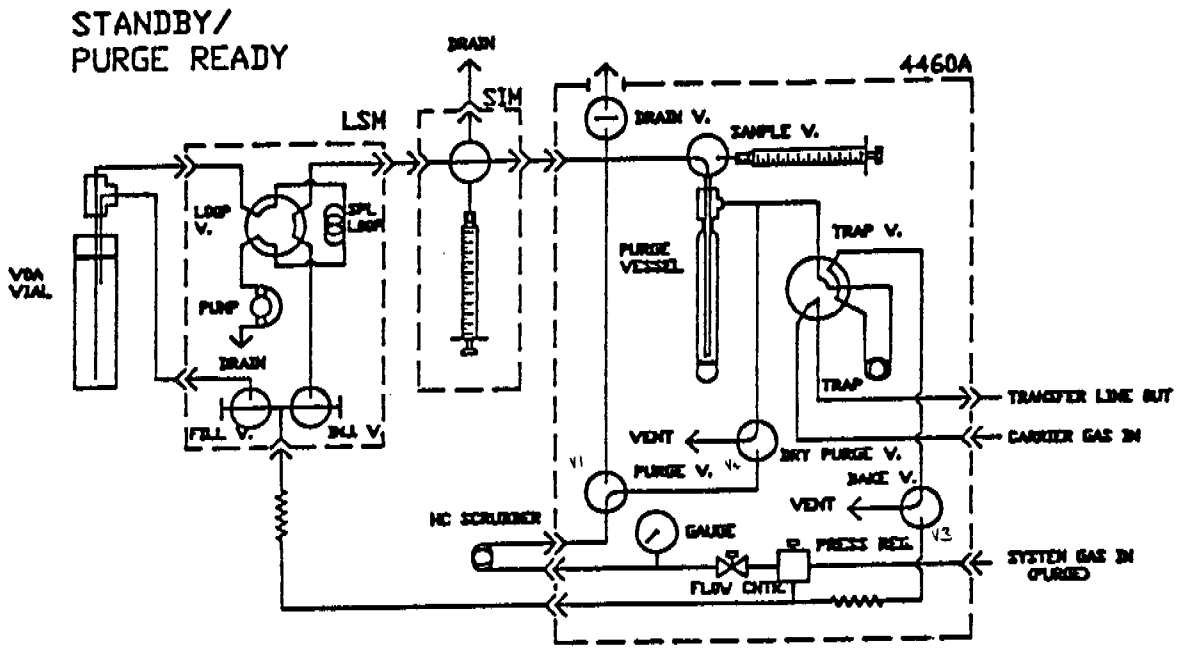
MANUAL DRAIN

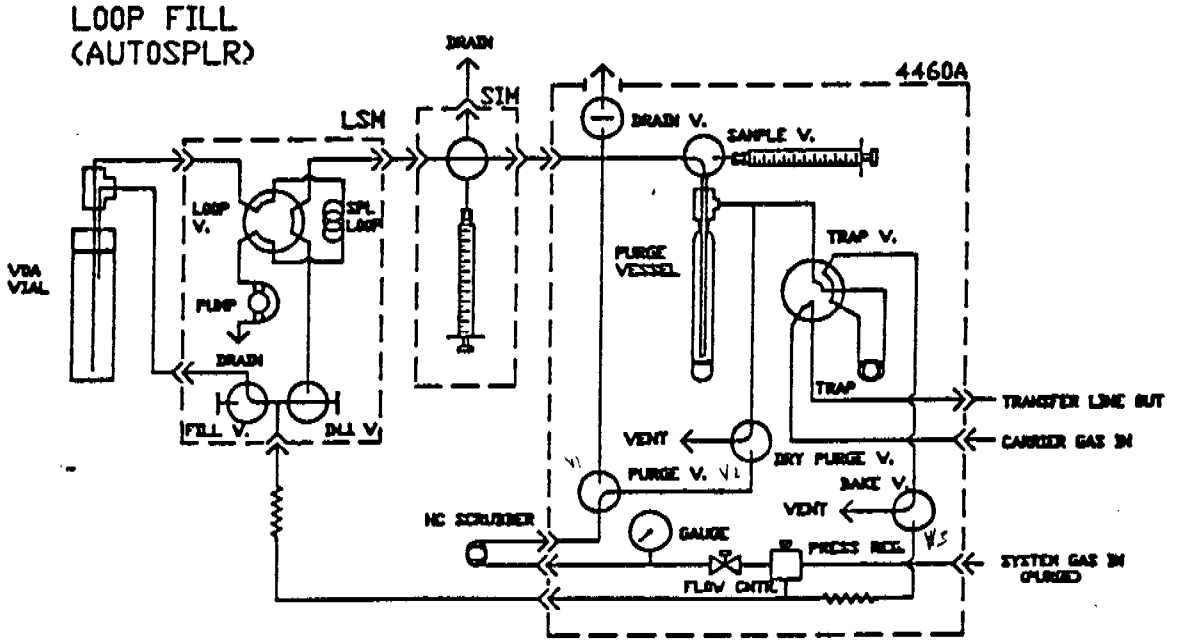


VALVES
BAKE
OUT-UP
DRY-OUT-UP
PURGE-IN-

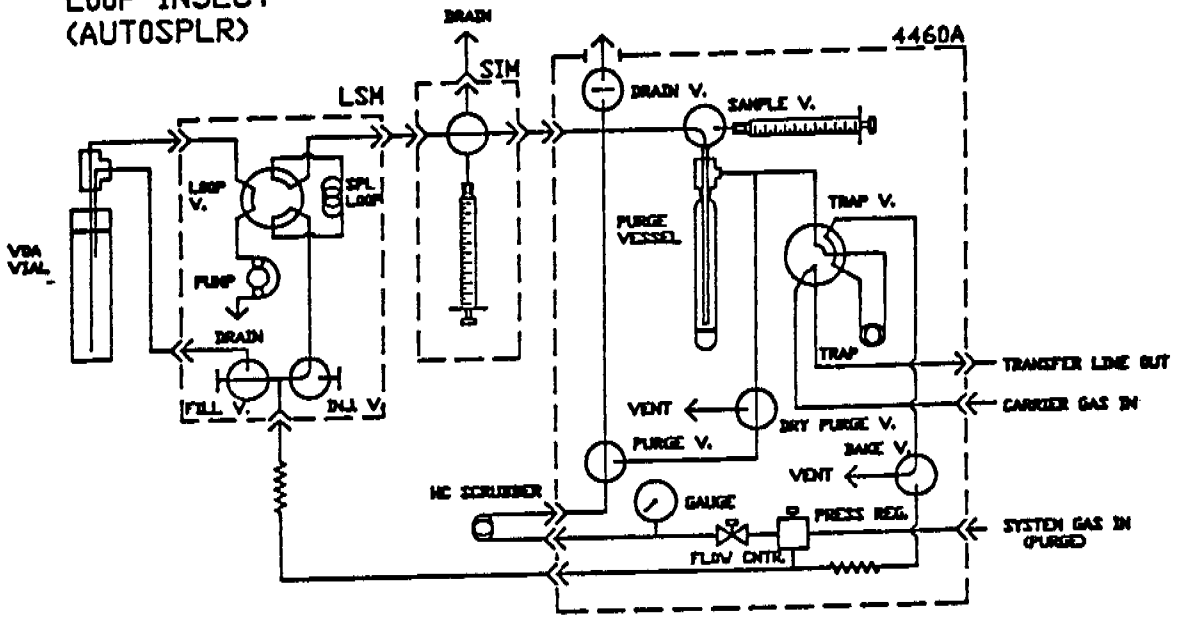
— CAP VALVE 88 DRY PURGE.
— 88 SCRUBBER

Needle Sparging

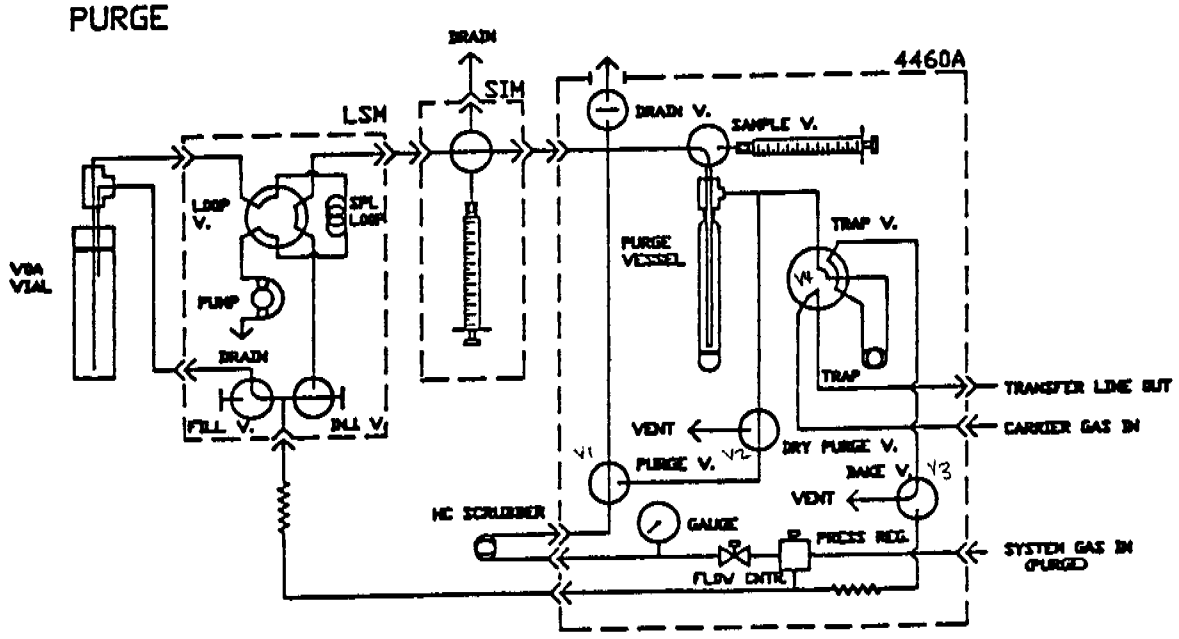




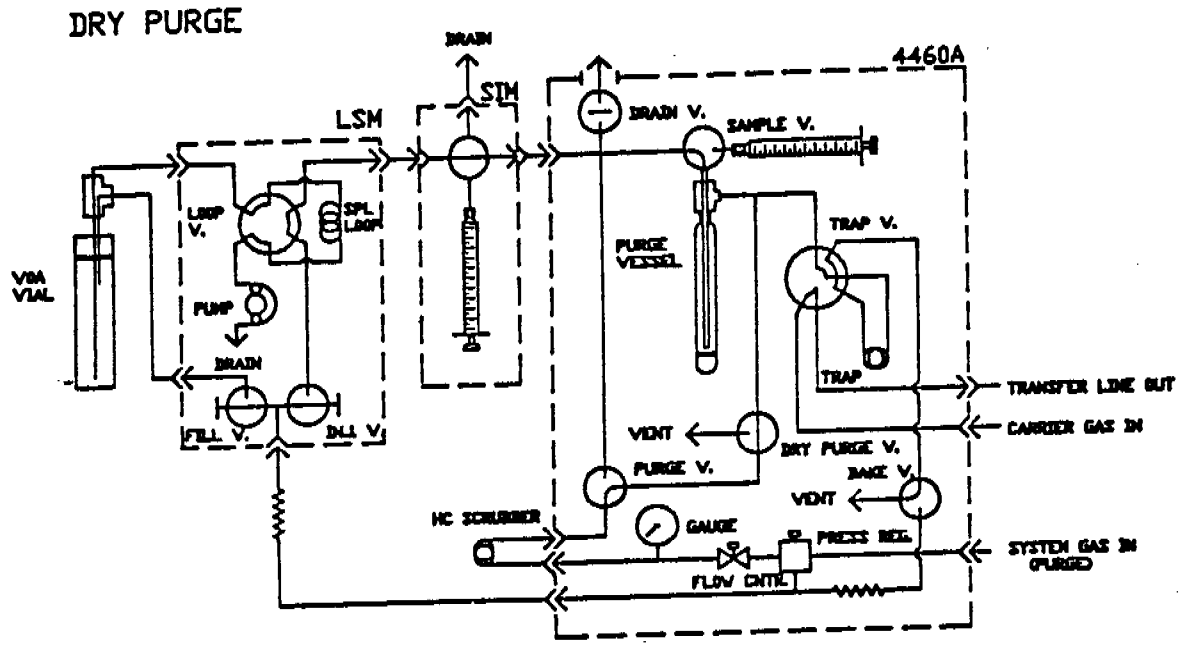
LOOP INJECT
(AUTOSPLR)

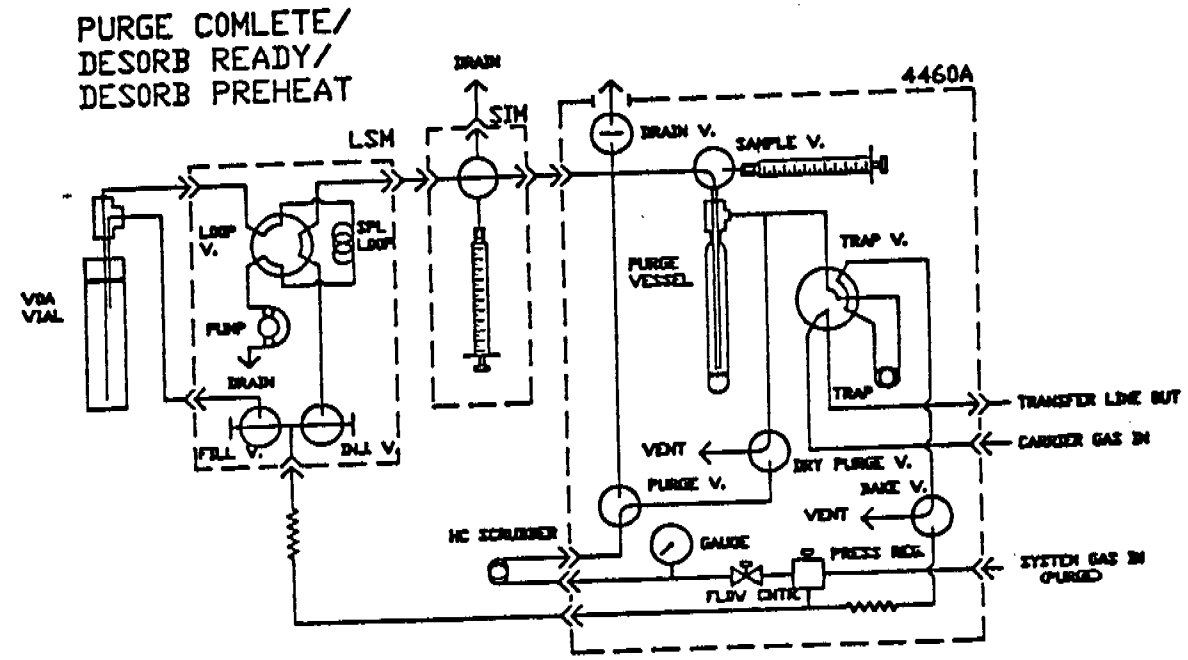


PURGE

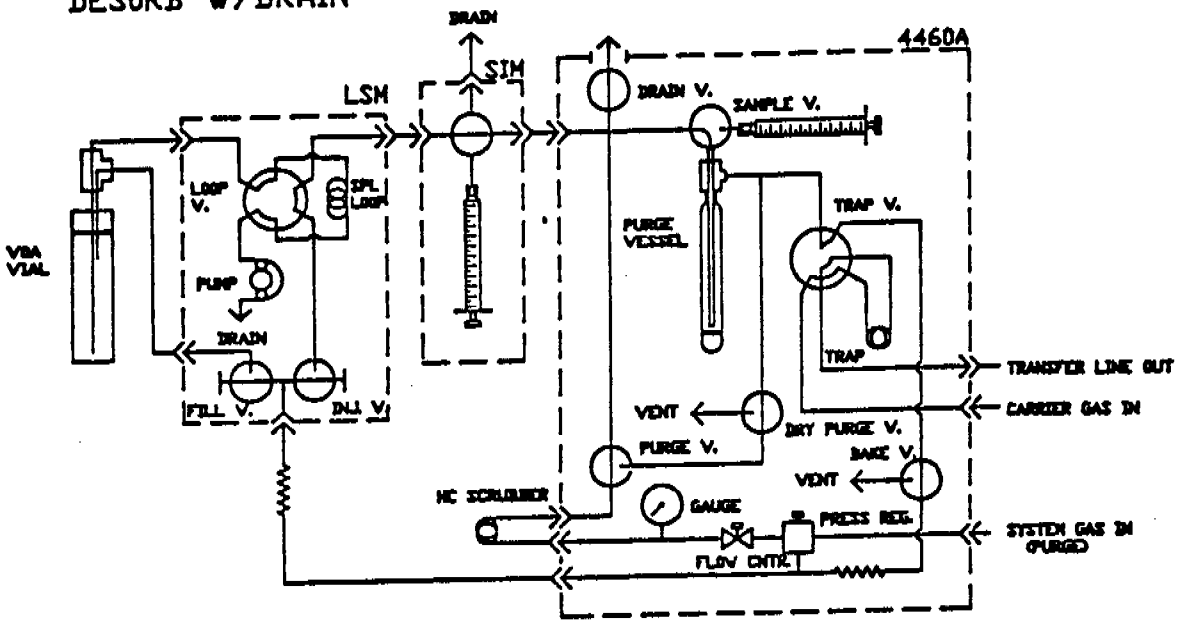


Needle Sparging

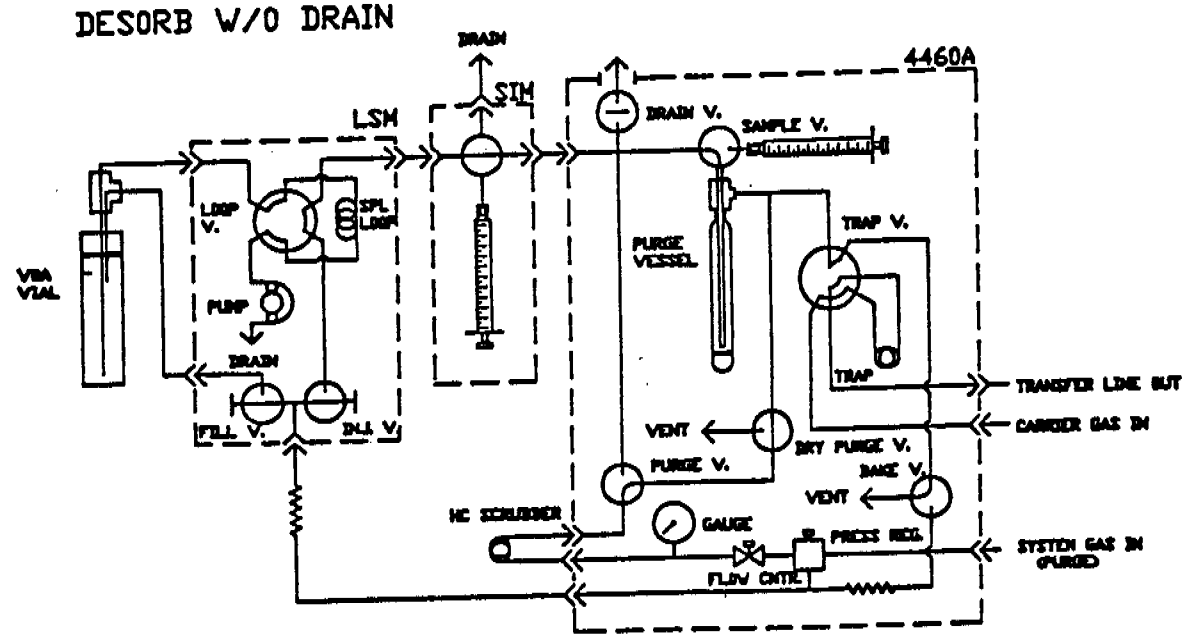




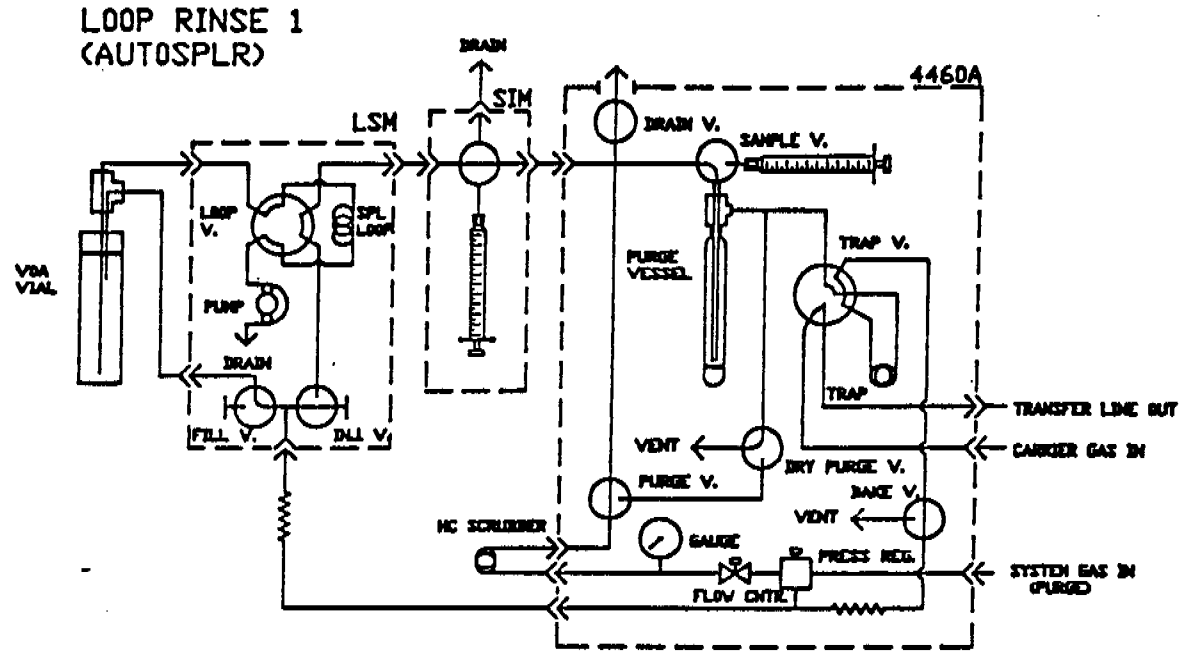
DESORB W/DRAIN

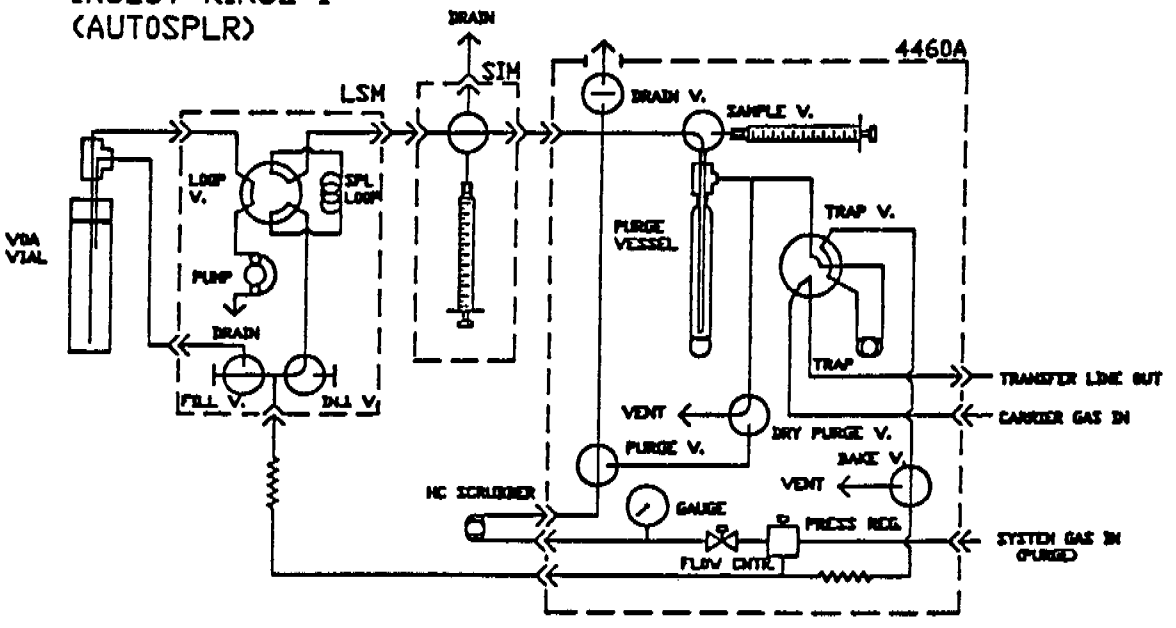


DESORB W/O DRAIN

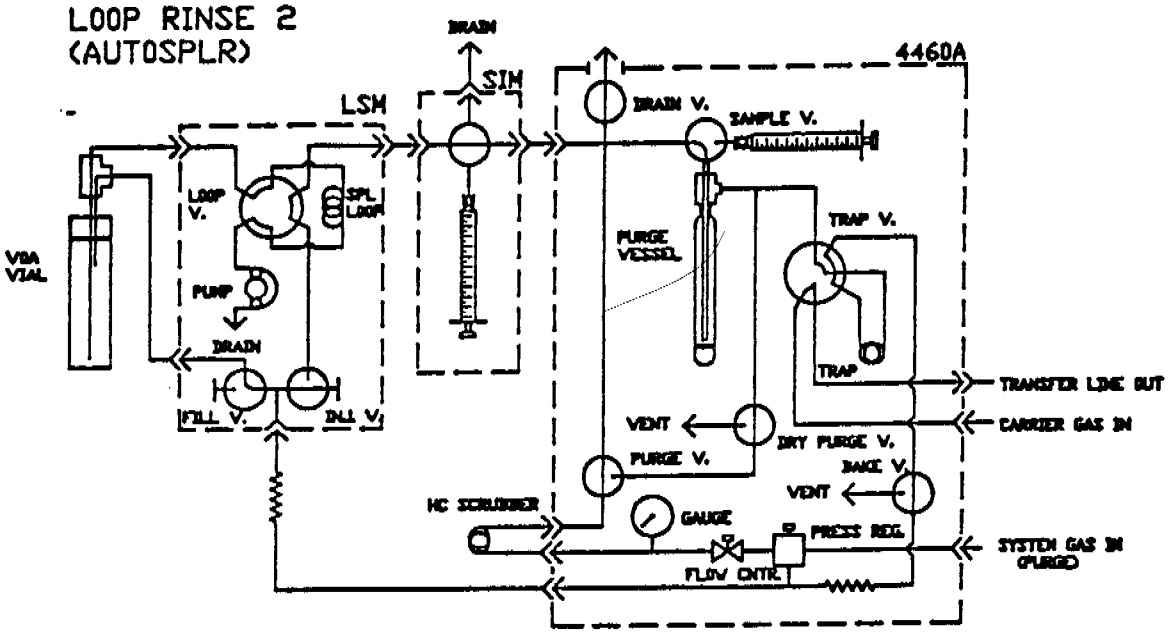


Needle Sparging

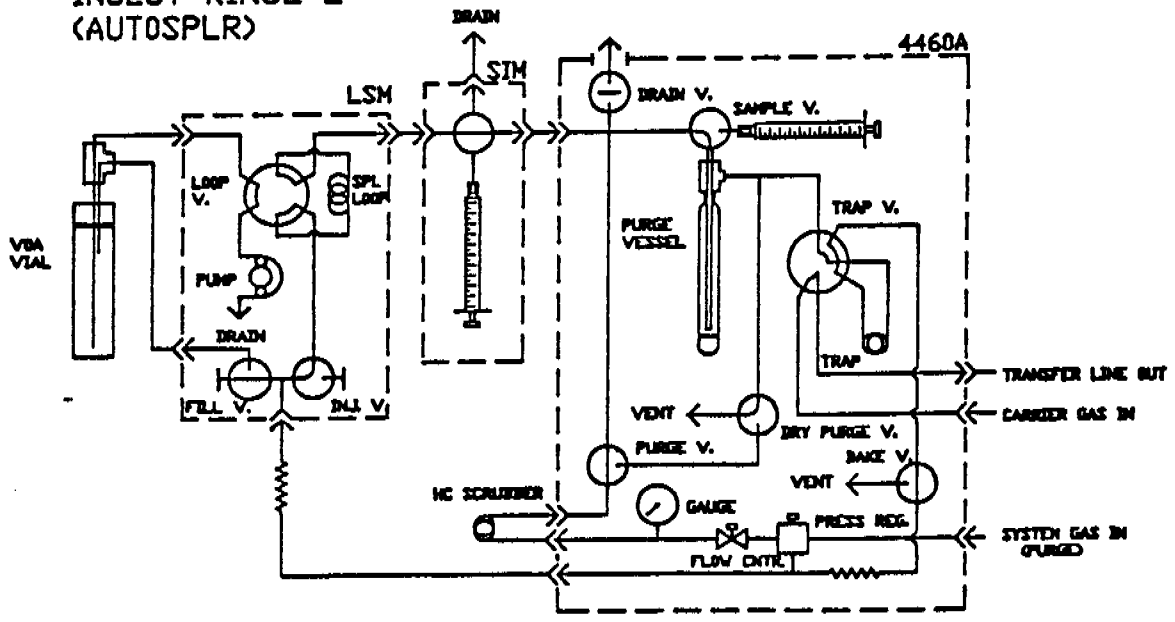




DRAIN RINSE 1/
LOOP RINSE 2
(AUTOSPLR)

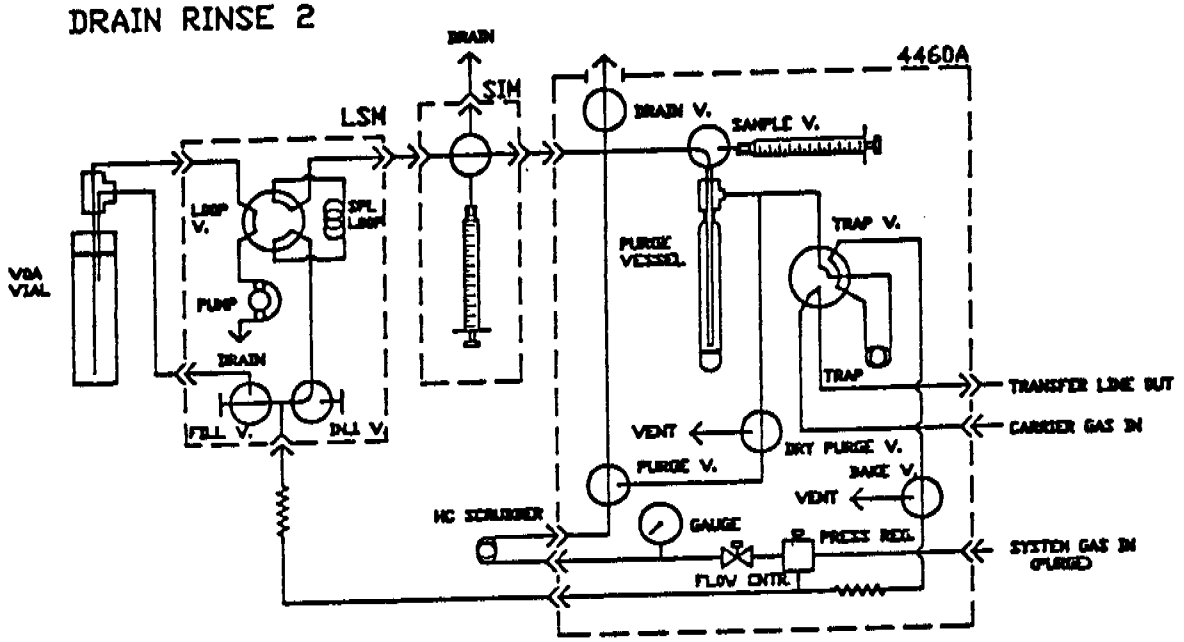


INJECT RINSE 2
(AUTOSPLR)

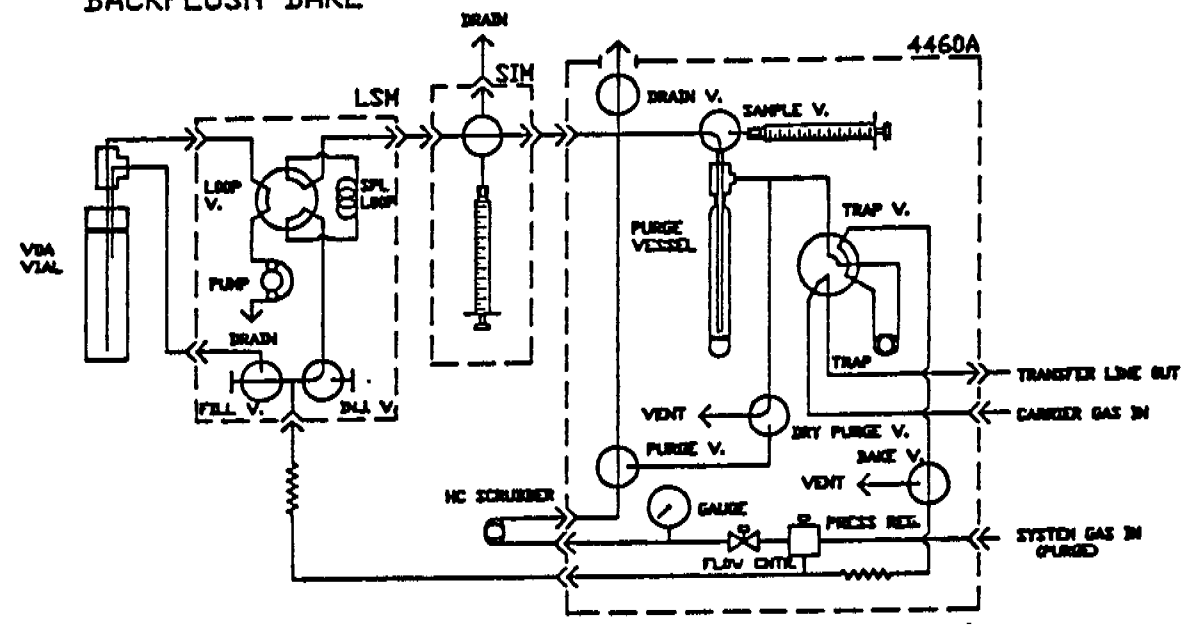


Needle Sparging

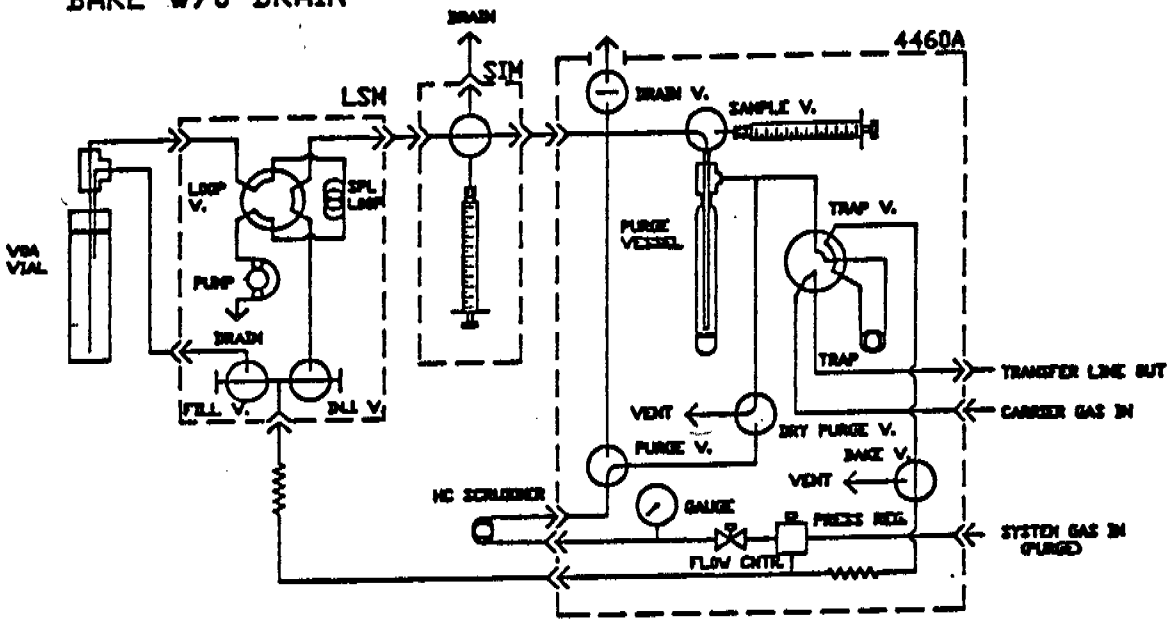
DRAIN RINSE 2



BACKFLUSH BAKE



BAKE W/O DRAIN



MANUAL DRAIN