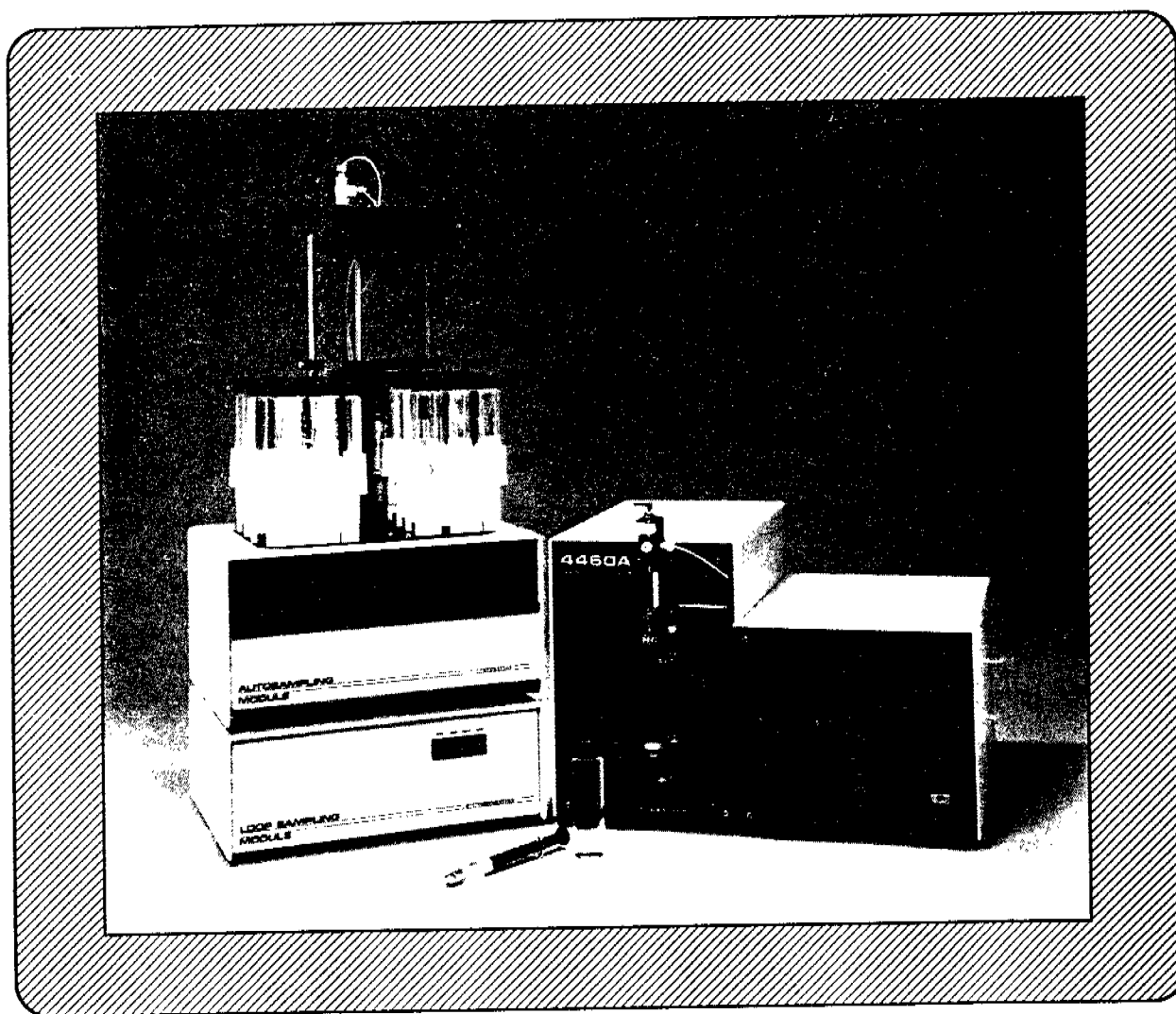


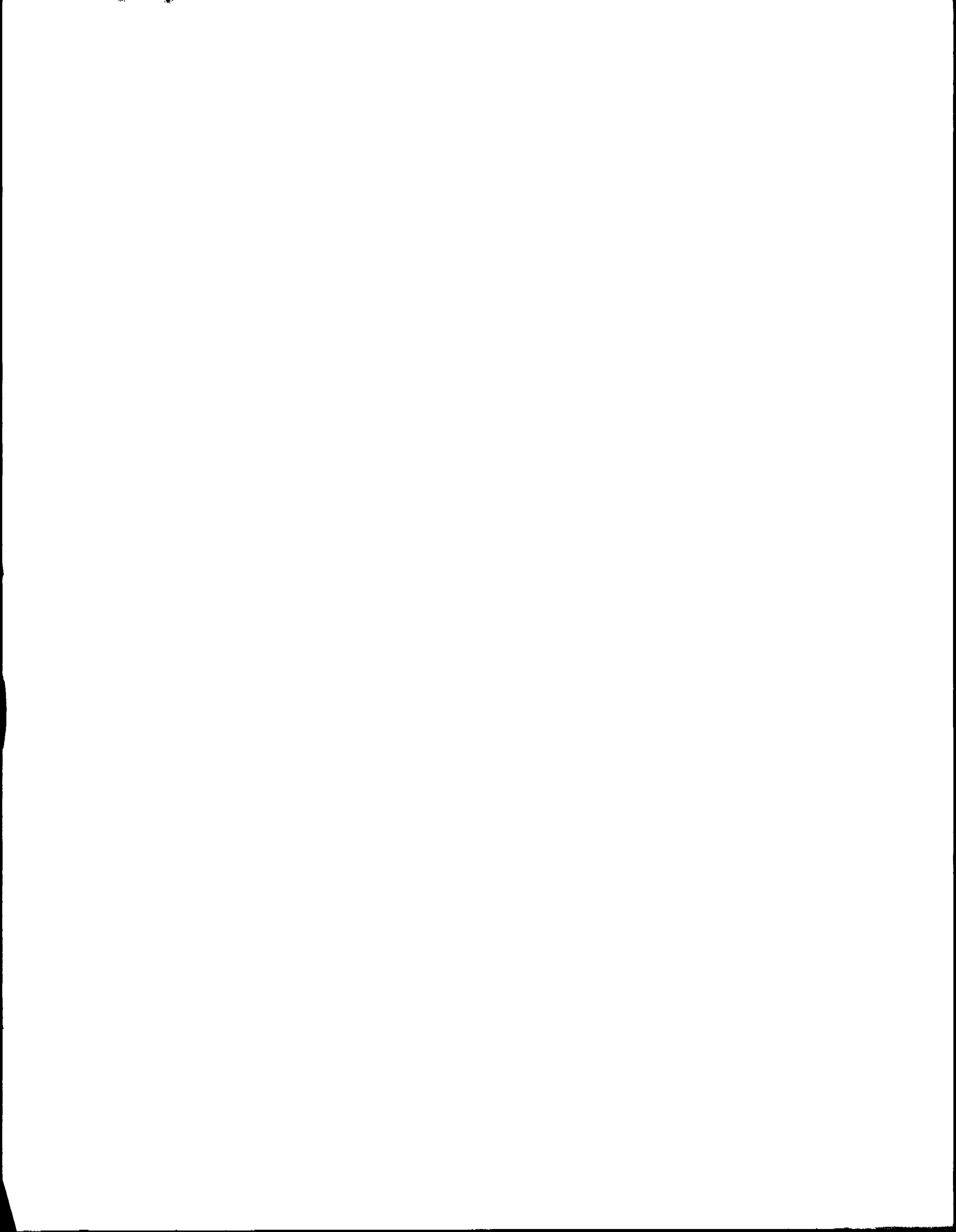
4460A Purge and Trap Concentrator

Operating and Service Procedures



O·I·CORPORATION

Graham Rd. at Wellborn Rd. • P.O. Box 2980 • College Station, Texas 77841-2980
Telephone (409) 690-1711 • FAX (409) 690-0440 • TWX No.:510-892-7944





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. Corporation. 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.

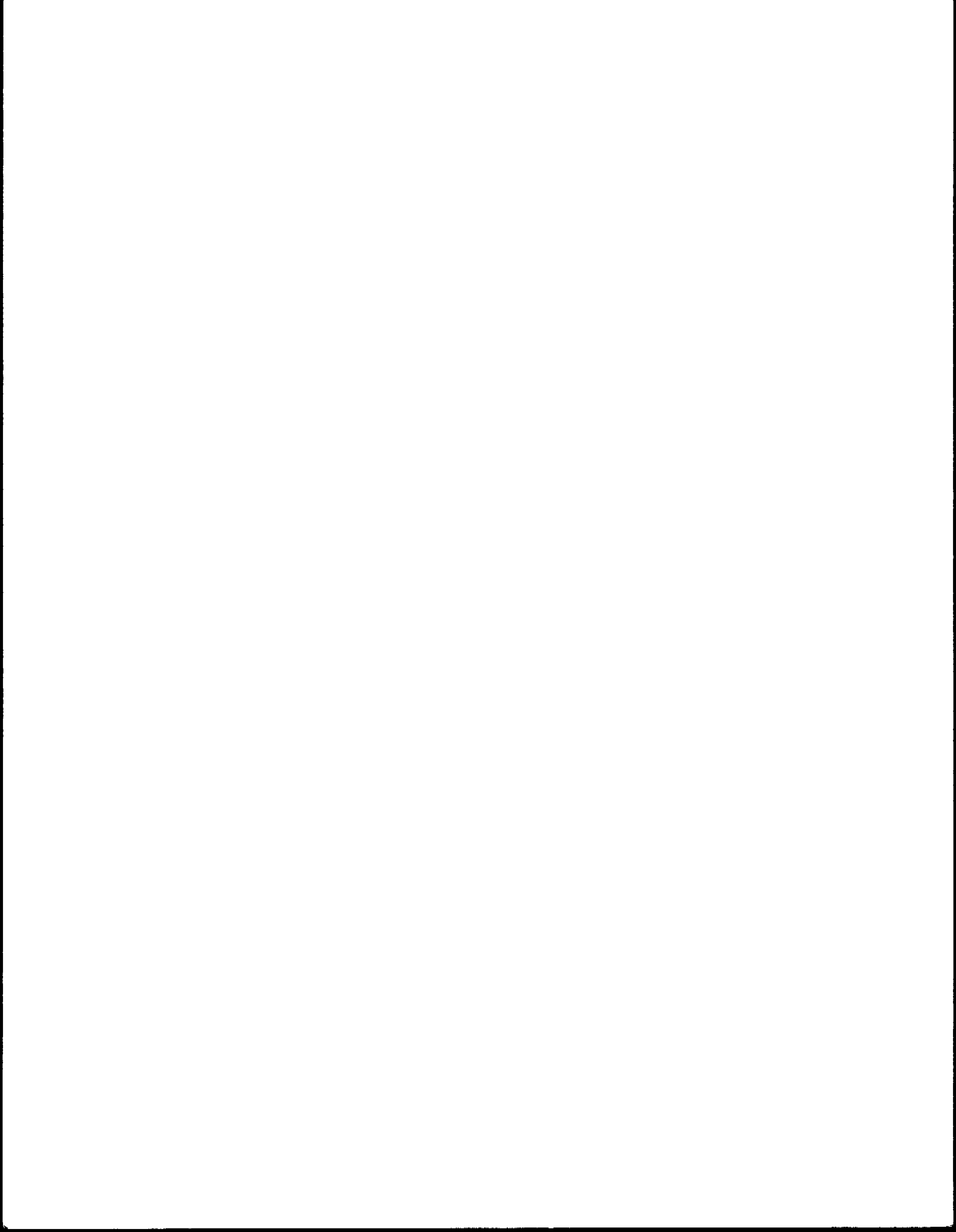


Table of Contents

Chapter 1 Model 4460A Sample Concentrator

1 Operational Concepts

- 1..... Instruments STATES
- 2..... Sequencing Through STATES
- 3..... Components Which Are Heated

3 Features

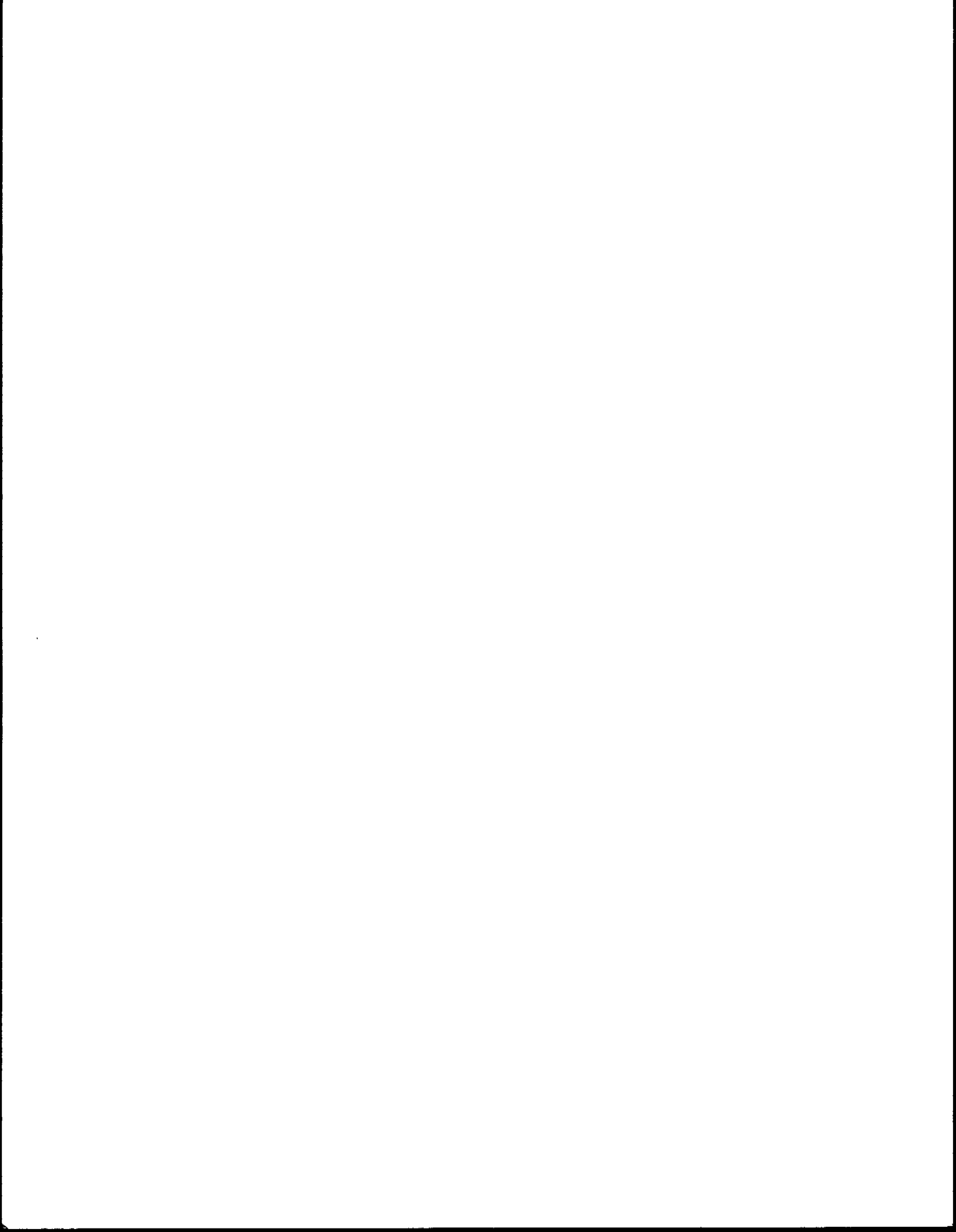
- 3..... Sample Concentrator
- 4..... VOA Vial Autosampler

4 Specifications

- 4..... Principle of Operation
- 4..... Physical Design
- 5..... Electrical Requirements
- 5..... Gas Requirements
- 5..... Traps
- 5..... Spargers
- 6..... STATES of Operation
- 6..... Time Functions
- 6..... Temperature Functions
- 7..... Autosampling Module
- 8..... Loop Sampling Module
- 9..... Standards Injection Module

10 Additional Useful Hardware

- 10..... Startup Kit
- 10..... Hookup Kit
- 10..... Syringe Kit
- 10..... GC Interface Kits



Chapter 2

Description of Components

13 Front Panel

13.....	Purge Vessel
13.....	Sample Vessel
13.....	Sample Valve
13.....	Tee Fitting
13.....	Syringe Port
14.....	Drain Line
14.....	Purge Gas Line
14.....	Septum Plug

15 Keys and Displays

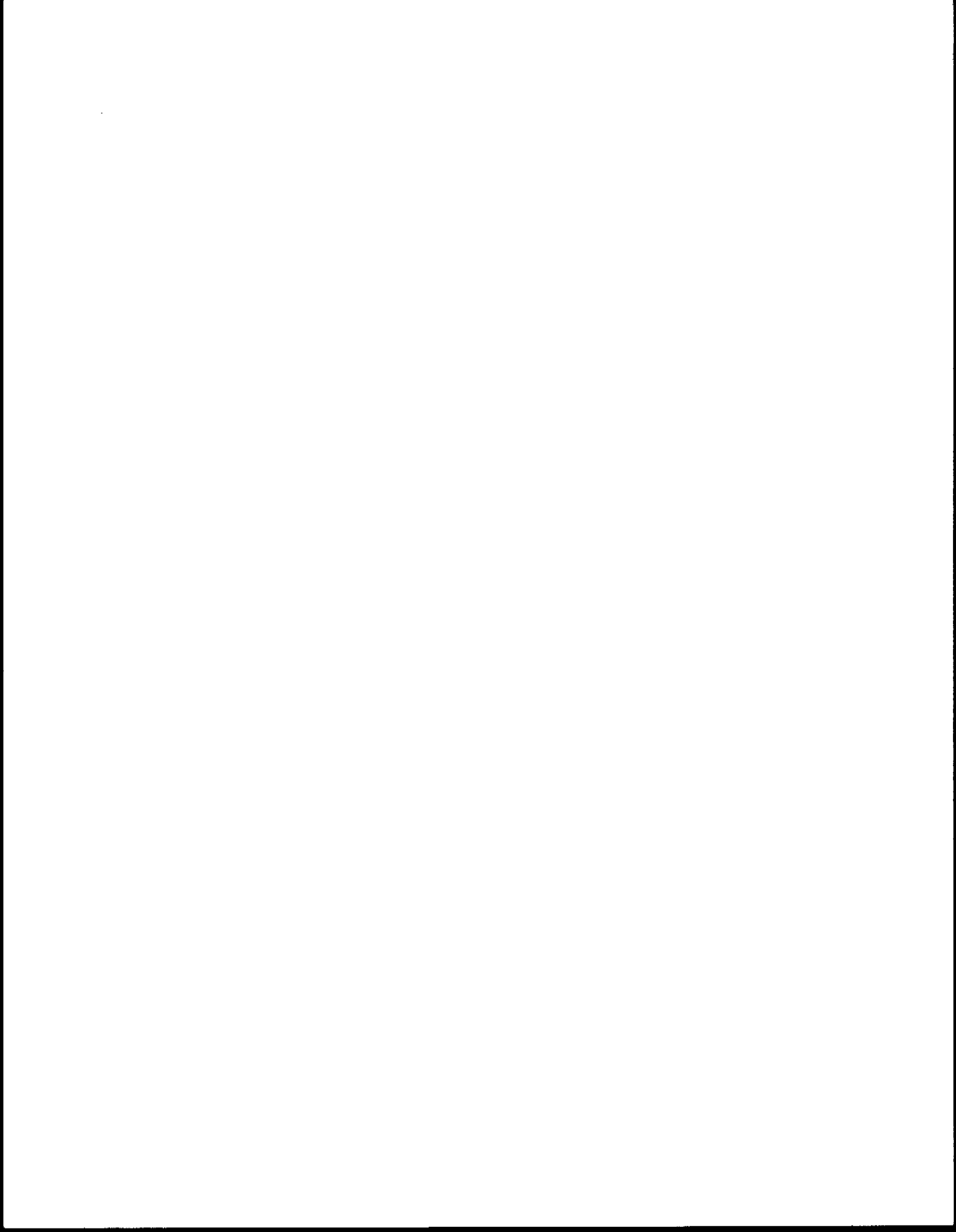
15.....	Power Switch
15.....	Time Display
15.....	Temperature Display
15.....	2nd FUNCT Switch
15.....	AUTO - ON/OFF Key
15.....	INC/DEC Switches
16.....	TIME Keys
17.....	TEMP Keys
18.....	AUTOSPL Keys
19.....	STATE STATUS LEDs
19.....	TRAP STATUS LEDs

19 Left Bay

19.....	Valve Oven
19.....	Trap
21.....	Transfer Line
21.....	Trap TC Receptacle
21.....	Xfer Line TC Receptacle
21.....	Xfer Line Power Receptacle
21.....	Fan 1 Power Receptacle
21.....	System Pressure Regulator
22.....	Purge Gas Flow Controller
22.....	Purge Gas Pressure Gauge
22.....	Trap Valve Actuator Solenoid
22.....	Trap Valve Actuator
22.....	Fan
22.....	Fan Duct Cover

23 Valve Oven

24.....	Trap Valve
24.....	Trap Bulkheads
24.....	Dry Purge Valve
24.....	Bake Valve
24.....	Purge Valve
24.....	Drain Valve



24 Right Bay

24.....	Program E/PROM
24.....	Microprocessor
24.....	Temperature Control Adjustment Pot
25.....	Dip Switches
26.....	Voltage Select Plug
26.....	DC Power Status LEDs
27.....	DC Control Status LEDs

27 Rear Panel

27.....	Purge Gas Inlet
27.....	Actuator Gas Inlet
27.....	Carrier Gas Inlet
27.....	Hydrocarbon Scrubber
27.....	Autosampler Gas Outlet
27.....	Transfer Line Feedthrough
28.....	Drain Line Feedthrough
28.....	Main AC Power Receptacle
28.....	Main AC Power Fuse
29.....	External Heated Zone Power Receptacle
29.....	Serial Port Receptacle
29.....	Autosampler Cable Receptacle
29.....	Computer Interface Receptacle
29.....	Terminal Strip - Relay I/O

30 Loop Sampling Module (LSM) - Front Panel

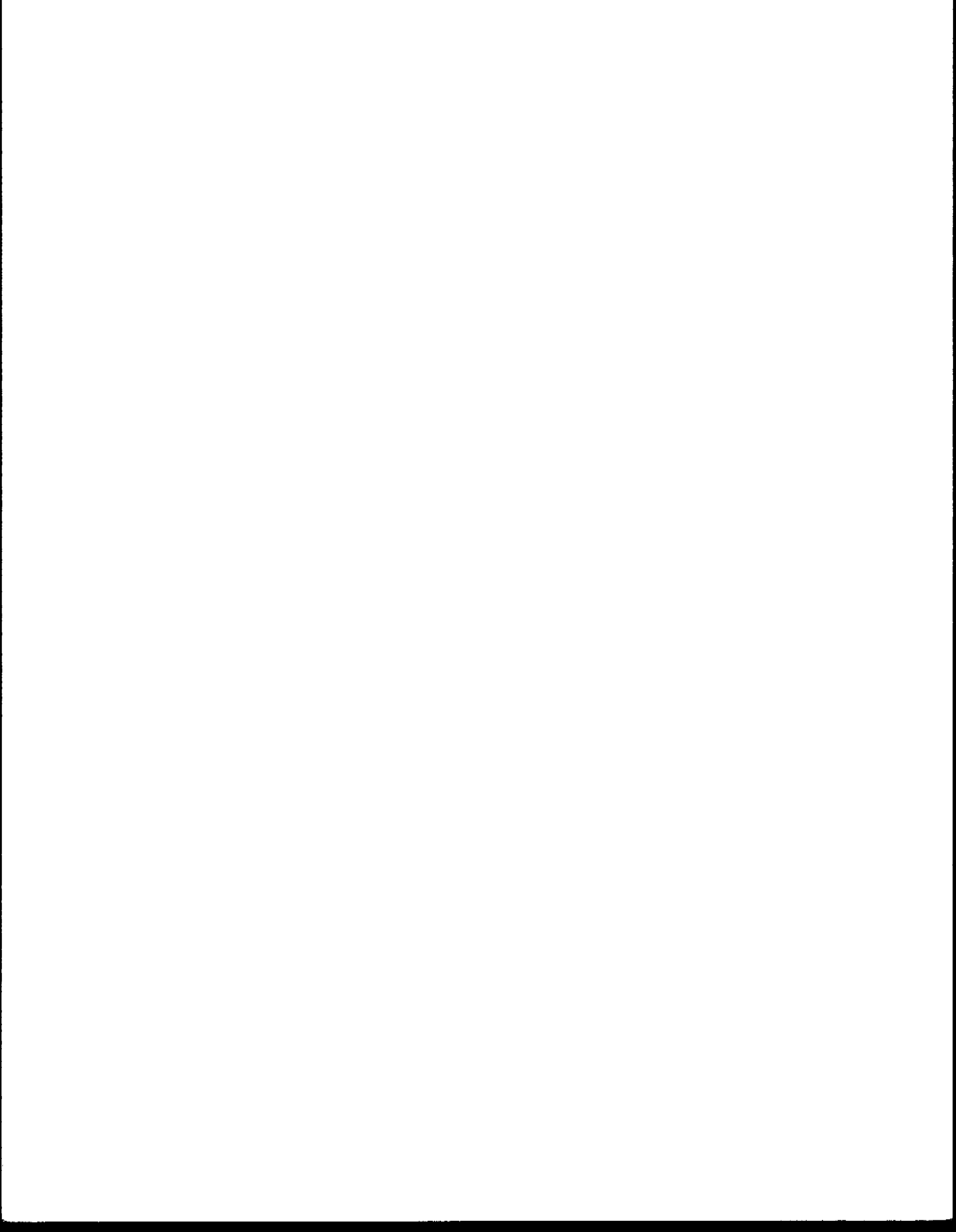
30.....	RAISE Key
30.....	LOWER Key
31.....	WASH Key
31.....	PUMP Key

32 Loop Sampling Module (LSM) - Rear Panel

32.....	LSM Power Receptacle
32.....	Power Output Receptacle
33.....	ASM Output Receptacle
33.....	Control Input Receptacle
33.....	LSM Sampler Inlet Line
33.....	LSM Fill Gas Outlet Line
33.....	Drain Line
33.....	LSM Sample Outlet Line
33.....	Sample Loop
33.....	LSM Fill Gas Inlet Line
33.....	LSM Actuator Gas Line

34 Loop Sampling Module (LSM) Interior

34.....	Sample Loop Valve
35.....	Sample Pump
35.....	Fill Gas Valve
35.....	Loop Inject Valve



35 Autosampling Module (ASM) - Front View

35.....	Rotary Tray
35.....	Vial Racks (Set of Nine)
35.....	Actuator Arm Assembly
35.....	Wash Station Assembly
35.....	Pipet Rod

36 Autosampling Module (ASM) - Rear View

36.....	ASM Power Receptacle
36.....	Manual Advance Button
37.....	Reset Button
37.....	Remote Input Receptacle
37.....	Sipper Output Receptacle
38.....	Hold-Down Foot Assembly
38.....	Wash Vessel
38.....	Septum Piercing Needle Assembly

38 Standards Injection Module (SIM) - Front View

38.....	ON/BYPASS Switch
39.....	MANUAL INJ Switch
39.....	SYRINGE ADV Switch
39.....	SYRINGE FILL Switch
39.....	SAMPLE OUT Port

39 Standards Injection Module (SIM) - Side View

39.....	Standards Syringe
39.....	Syringe Mounting Block
39.....	Syringe Plunger Arm

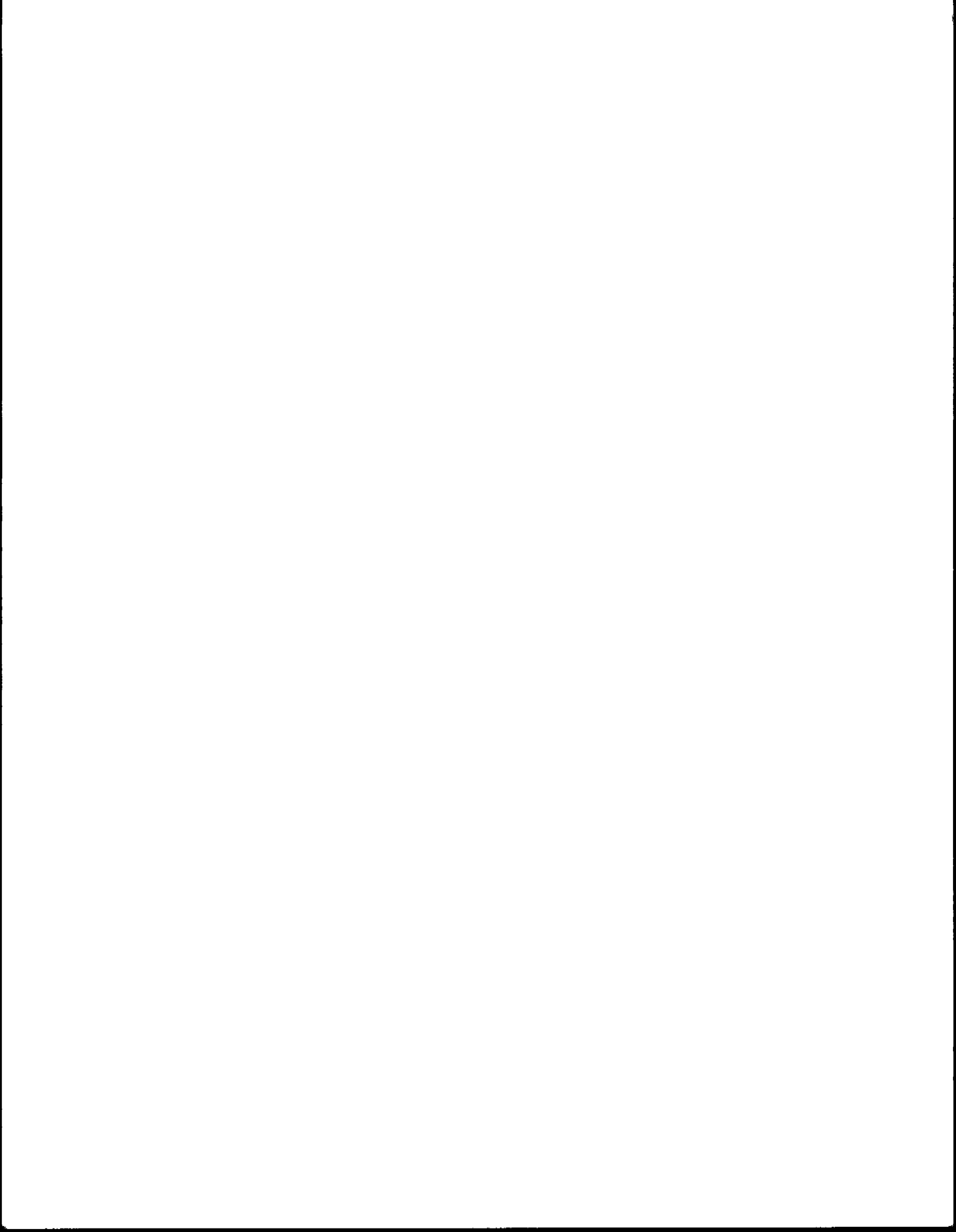
40 Standards Injection Module (SIM) - Rear View

40.....	SPL IN Port
40.....	SPL DRAIN Port
41.....	INJ MULTIPLIER Switch
41.....	IN Receptacle
41.....	OUT Receptacle

Chapter 3 Installation

43 Materials Needed

43.....	Purge Gas Source
43.....	Carrier Gas Source
43.....	Actuator Gas Source
44.....	Gas Hookup Lines
44.....	GC Interface Kit
44.....	Power Source



44 4460A Setup

44.....	Unpacking/Positioning the Instrument
44.....	Gas Inlet Connections
46.....	Transfer Line and Drain Connections
47.....	Sample Valve and Purge Vessel Assembly
48.....	Trap Installation
49.....	Standard Carrier Interface Connections
50.....	Low-Dead-Volume Interface Connections
52.....	Electrical Connections

53 Autosampler System Setup

53.....	Autosampling Module (ASM) Assembly
55.....	Tubing Connections
57.....	Electrical Connections

57 Setup of SIM with 4460A and LSM

57.....	Tubing Connections
57.....	Electrical Connections

57 Needle Sparging Setup

57.....	Needle Sparging Assembly
---------	--------------------------

Chapter 4 Operation

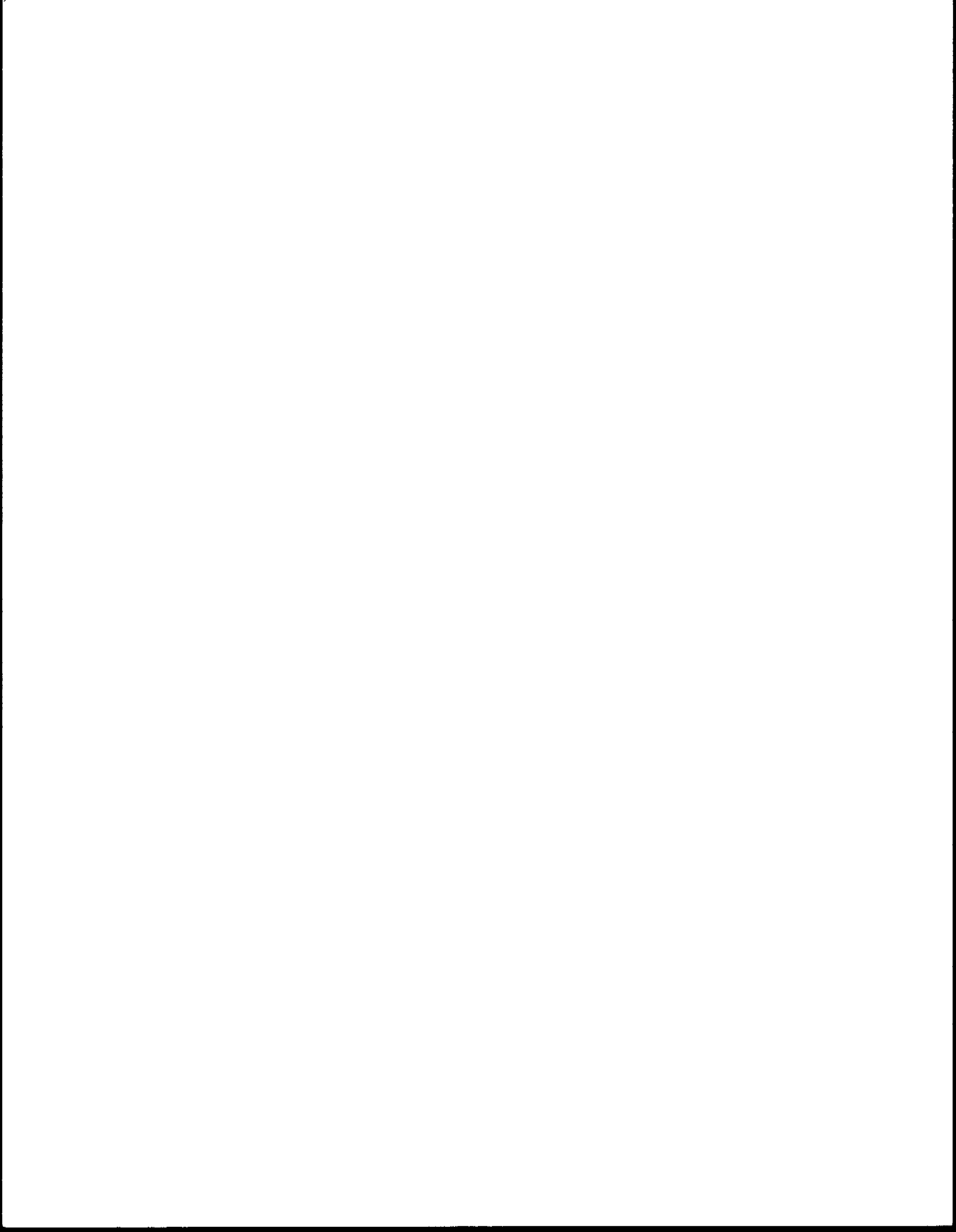
61 Overview of Method

61.....	Sample Introduction
61.....	4460A Syringe Injection
62.....	Autosampler Injection

65 4460A Functional Checks

65.....	Pre-Power-Up Checks
65.....	Confirming Trap Valve Rotation
66.....	Setting Purge Gas Flow Rate
67.....	Purge Gas Leak Check
67.....	Drain Check
67.....	Carrier Gas Leak Check
68.....	Trap Bake

68 Autosampling System Functional Checks



69 Running A Sample

69.....	Syringe Sample Loading
70.....	Syringe Sample Injection
70.....	Use of Manual DRAIN State
70.....	Use of CLEAR Command
70.....	Use of DESORB W/O DRAIN State

71 Autosampler Operation

71.....	Setting Autosampler Time Functions
71.....	Instructions for Operation

71 Standards Injection Module Operation

71.....	Principle of Operation
72.....	Pre-Operational Steps
72.....	Running an Analysis

72 Needle Sparging

73.....	Instructions for Needle Sparging Liquid Samples
73.....	Instructions for Needle Sparging Solid or Sludge Sample

Chapter 5

Maintenance and Troubleshooting

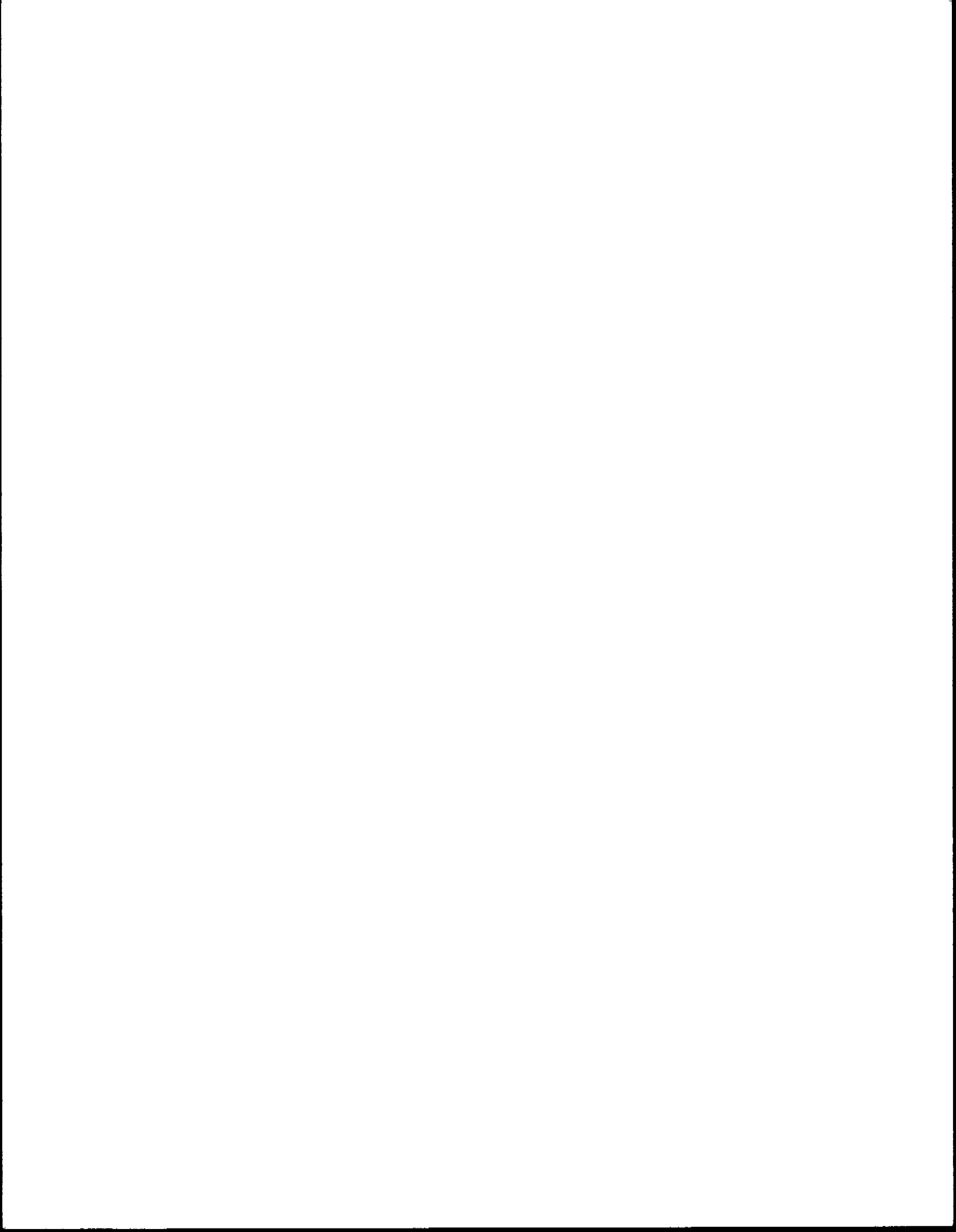
75 Trap Maintenance

75 4460A Diagnostic Tools

75.....	Manual Actuator Button
76.....	Purge Flow Controller/Pressure Gauge
77.....	Carrier Flow Controller/Pressure Gauge

77 Troubleshooting

77.....	4460A
77.....	Autosampling System



Chapter 6 Replacement Parts

79 Parts For 4460A

79.....	Boards
80.....	Ferrules
80.....	Fittings
80.....	Interface Assemblies
81.....	Support Items
81.....	Traps
81.....	Valves
81.....	Needle Sparging

82 Parts For Autosampling System

82.....	ASM Parts
82.....	LSM Parts

Chapter 7 Flow Schematics

86 Frit Sparging

94 Needle Sparging

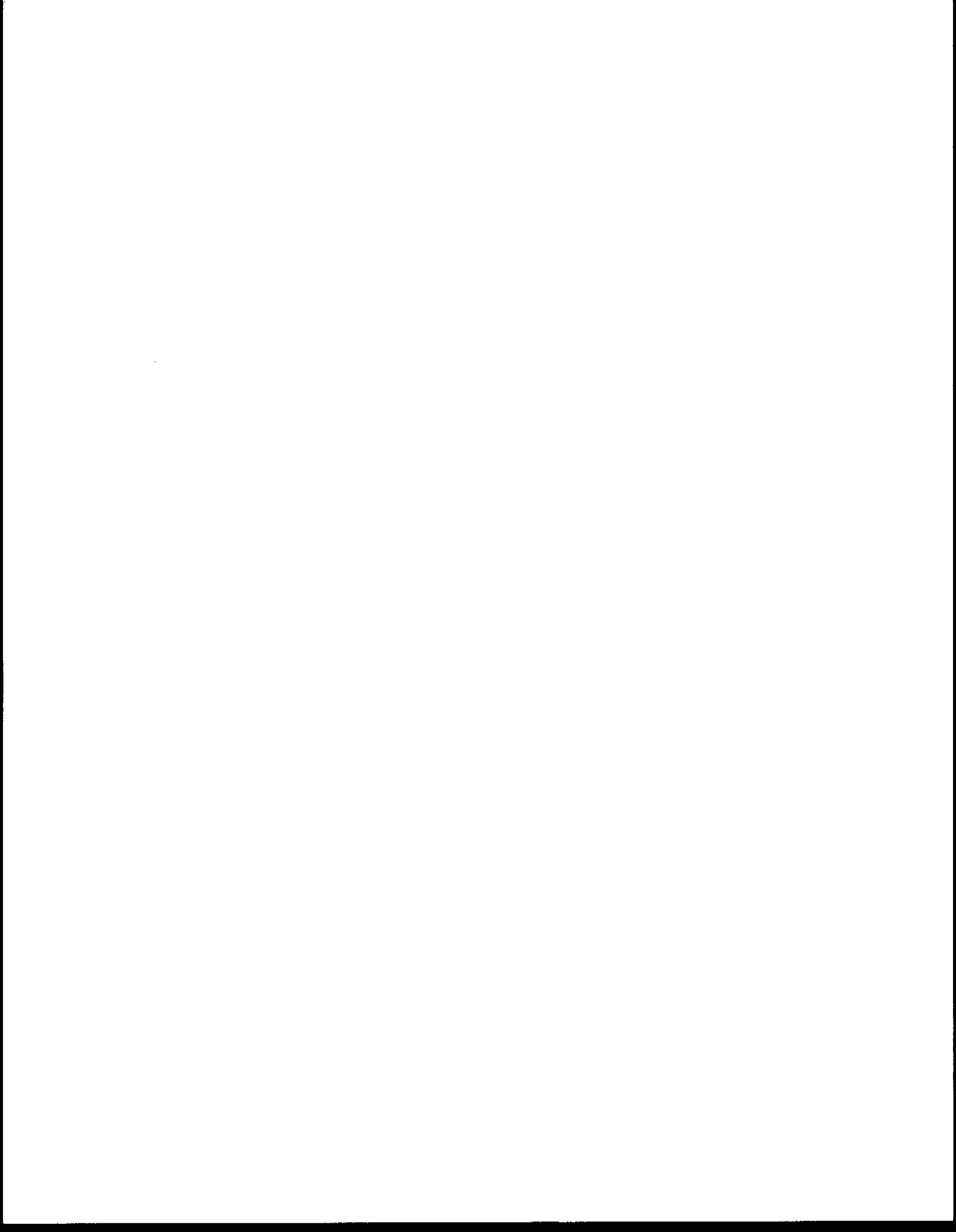
Descriptive Figures (Chapter 2)

14 4460A

14.....	Front Panel
16.....	Keys and Displays
20.....	Left Bay
23.....	Valve Oven
26.....	Right Bay
28.....	Rear Panel

30 LoopSampling Module (LSM)

31.....	Loop Sampling Module - Front Panel
32.....	Loop Sampling Module - Rear Panel
34.....	Loop Sampling Module - Interior



35 AutoSampling Module (ASM)

- 36..... Autosampling Module - Front View
- 37..... Autosampling Module - Rear View

38 Standards Injection Module (SIM)

- 38..... Standards Injection Module- Front View
- 40..... Standards Injection Module - Side View
- 41..... Standards Injection Module - Rear View

Installation Figures (Chapter 3)

44 4460A Setup

- 45..... Gas Inlet Connections
- 46..... Transfer Line and Drain Connections
- 47..... Assembly of Sample Valve and
Purge Vessel Components
- 48..... Trap Installation
- 49..... Standard Carrier Interface Connections
- 51..... Standard Low-Dead-Volume Interface
Connections
- 53..... Electrical Connections

53 Autosampler System Setup

- 54..... Autosampling Module (ASM) Assembly
- 55..... Tubing Connections
- 56..... Electrical Connections

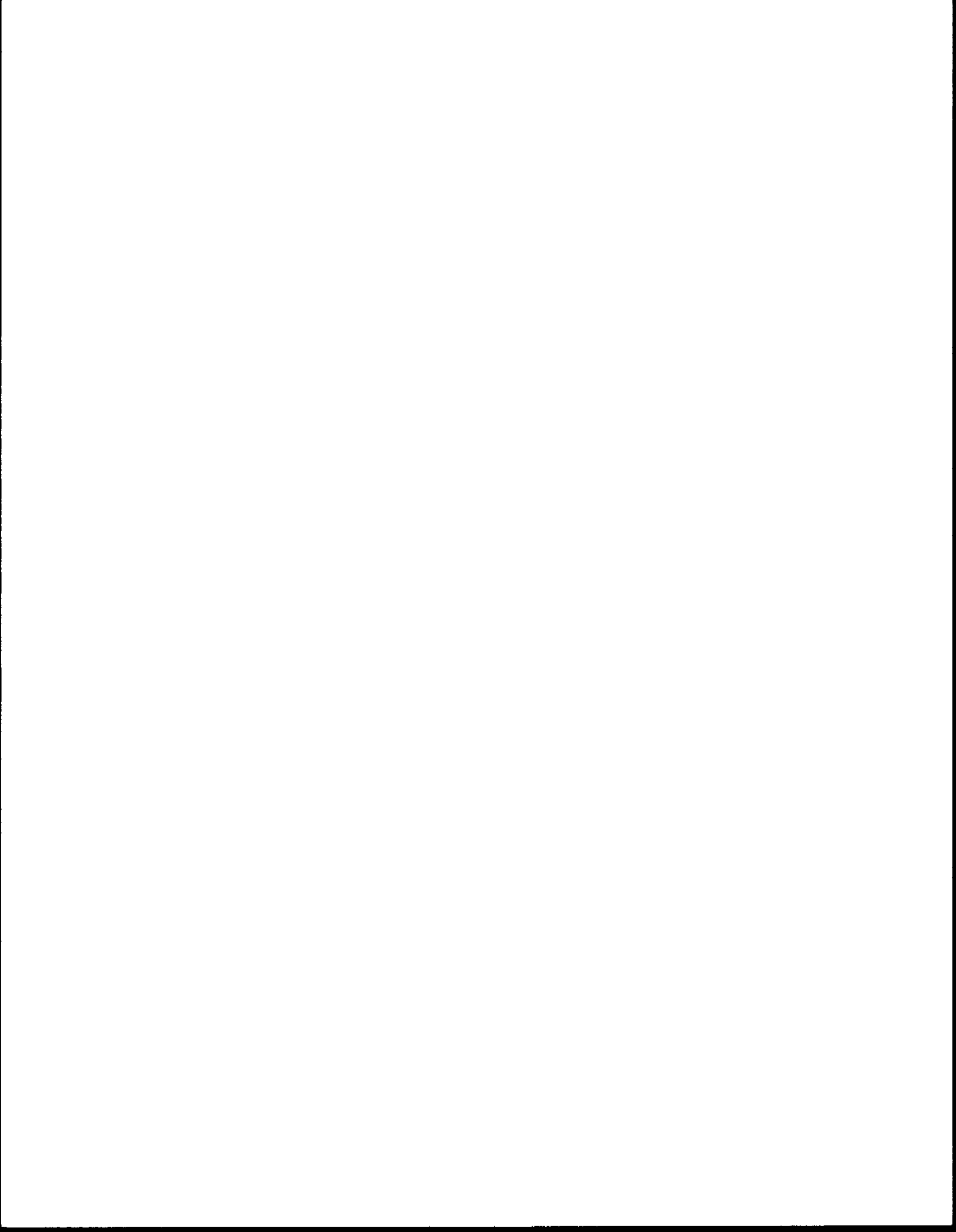
57 Needle Sparging Assembly

- 58..... Needle Sparging Connections
- 59..... Needle Sparging Connections (Detail 1)
- 59..... Needle Sparging Connections (Detail 2)
- 59..... Needle Sparging Connections (Detail 3)

Operation Figures (Chapter 4)

59 4460A

- 61..... Purge Ready
- 62..... Syringe Sample Injection
- 62..... Purge
- 62..... Dry Purge
- 63..... Purge Complete/Desorb Complete/
Desorb Preheat
- 63..... Desorb w/Drain
- 63..... Backflush Bake



64 Autosampler

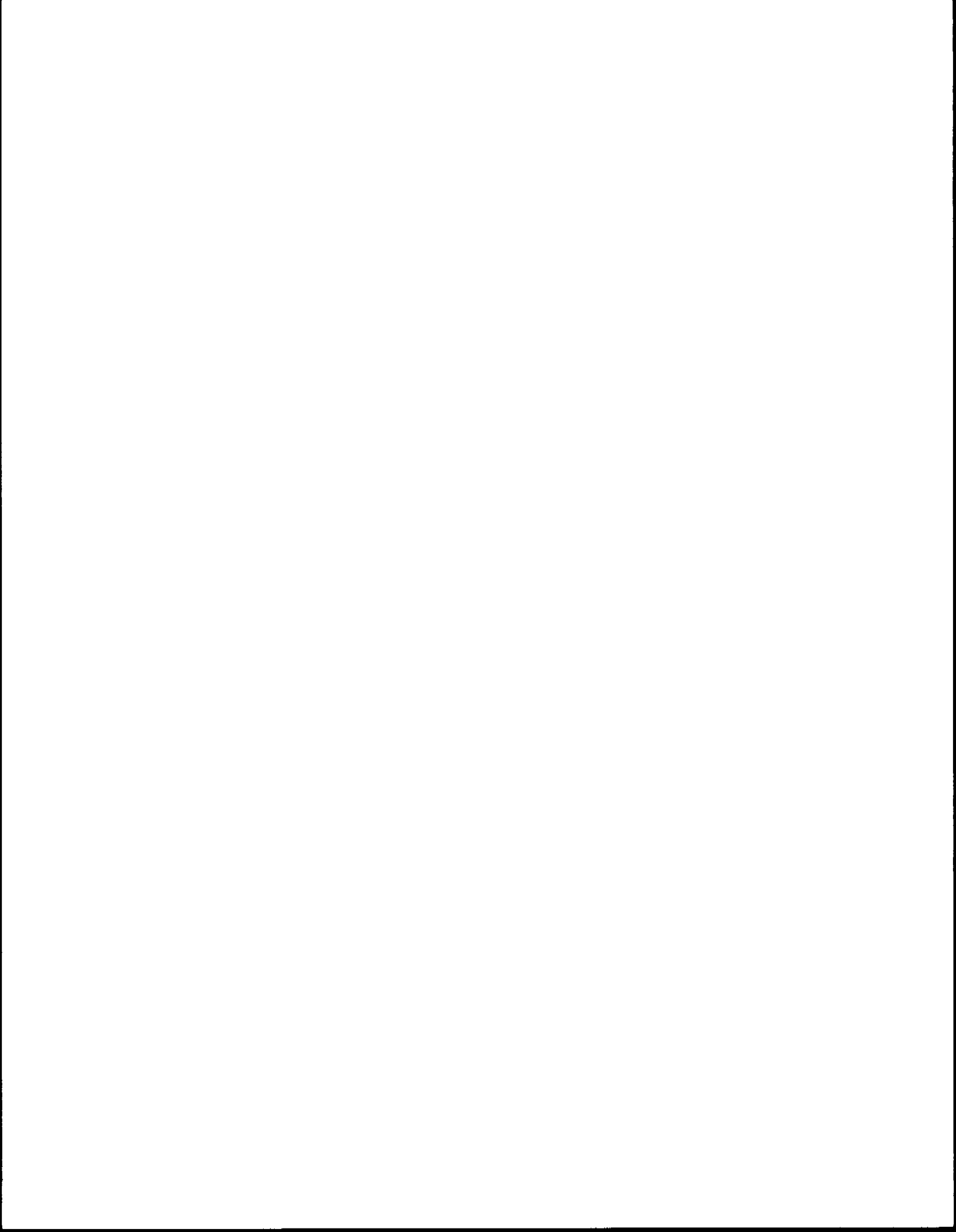
- 64..... Loop Fill
- 64..... Loop Inject
- 64..... Loop Rinse

65 4460A Functional Checks

- 65..... Sample Valve Drain vs. Fill Positions
- 66..... Setting Purge Gas Flow Rate

70 Autosampler System Functional Checks

- 70..... Syringe Sample Injection



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 auto-sampler 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

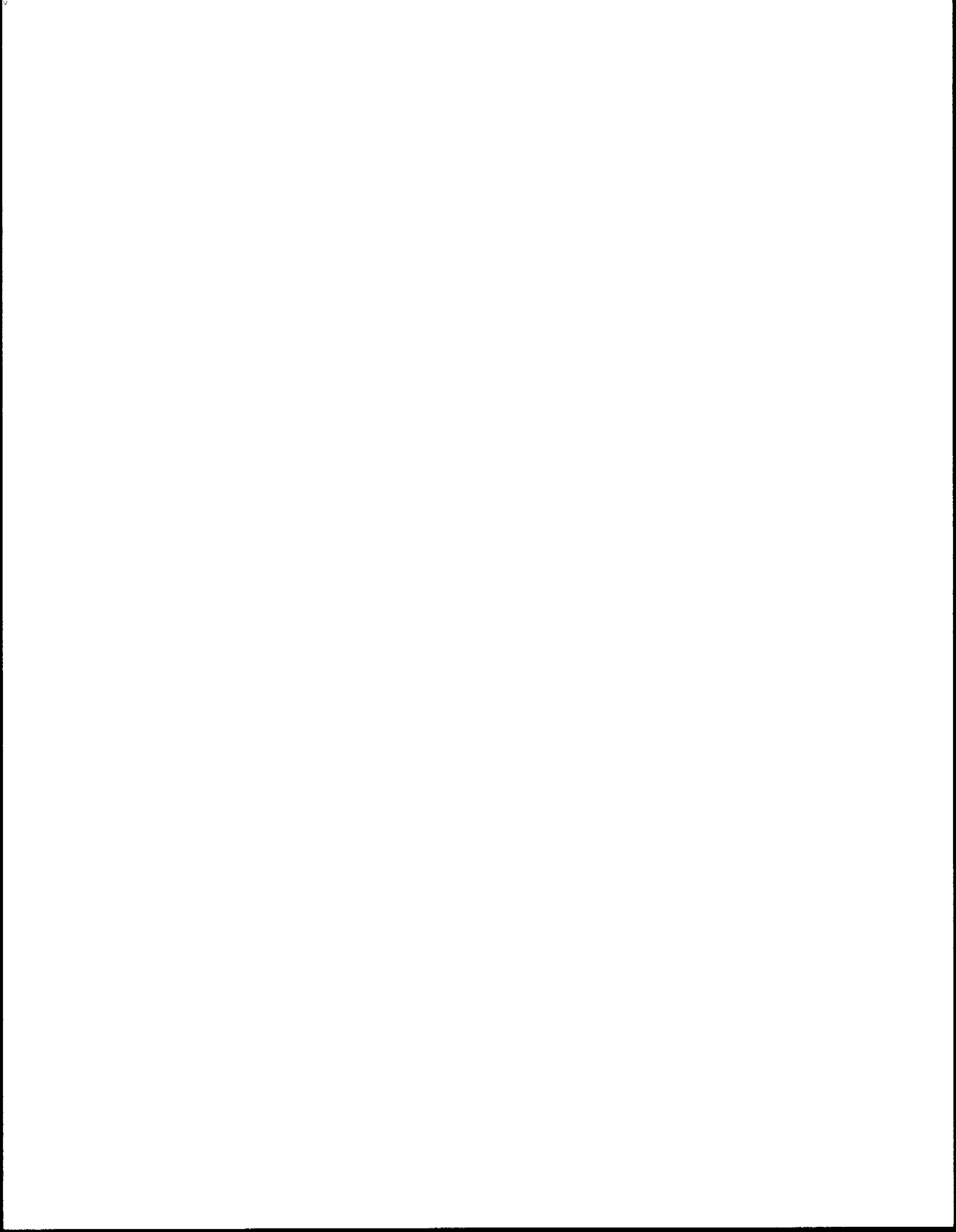
The various sets of valve and trap temperature combinations defined for analysis using the 4460A are called **states**. The eleven primary states are called:

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:

LOOPFILL	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

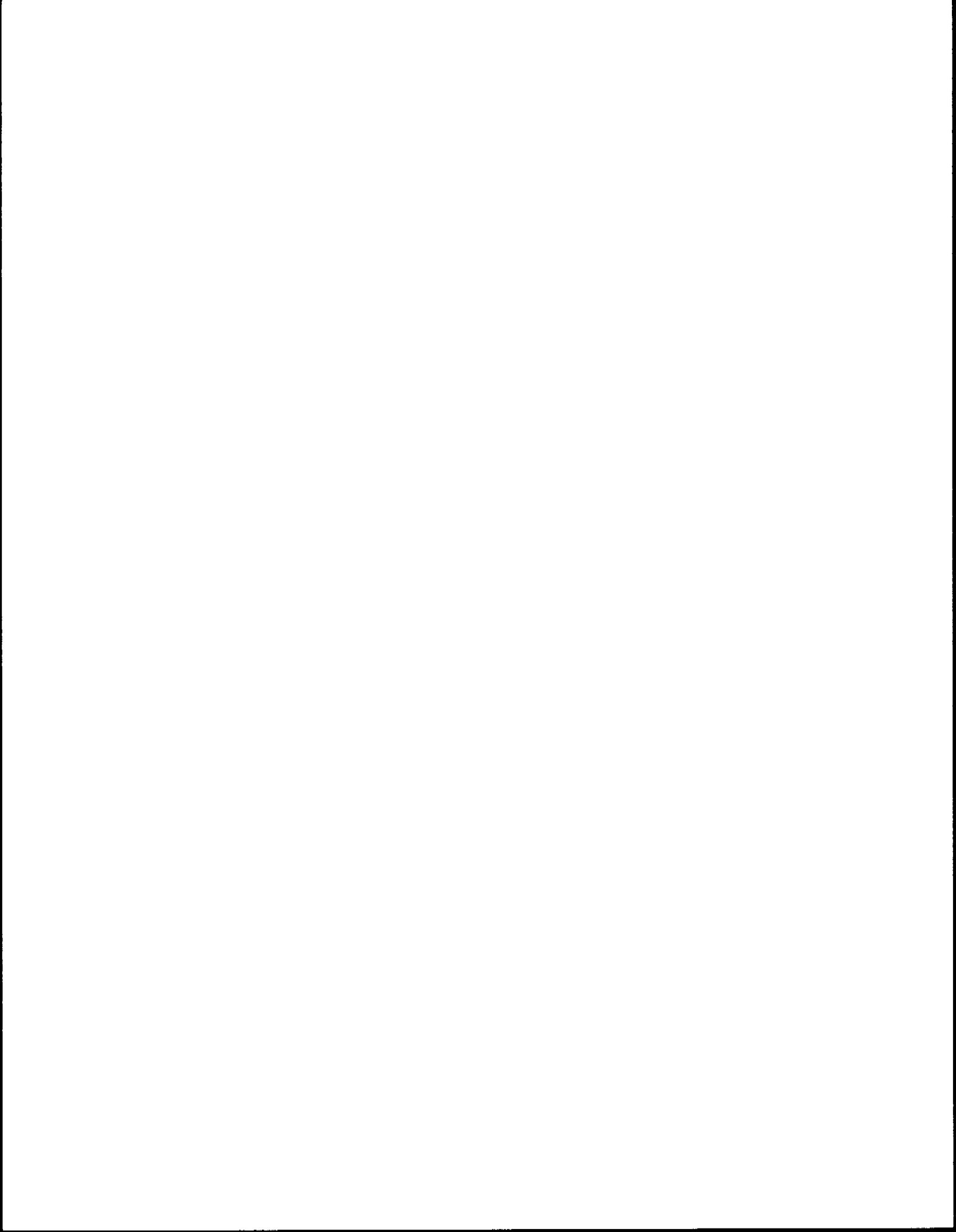
The states DRAIN and DESORB W/O DRAIN are not in the automatic sequence, but can be manually set.

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:

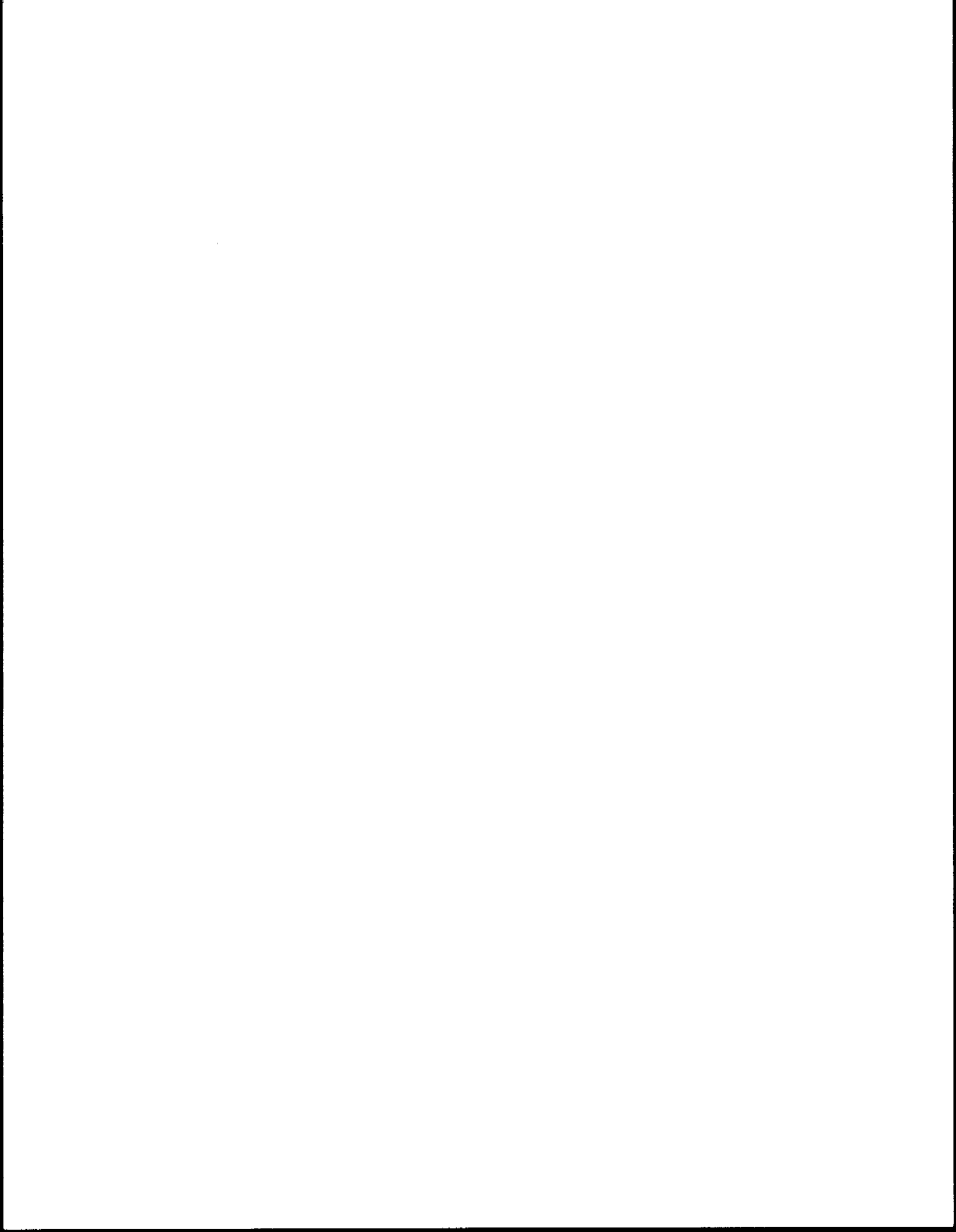
Trap: Purge Temp	Valve Oven
Desorb Temp	Transfer Line
Bake Temp	External Heated Device

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 than 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

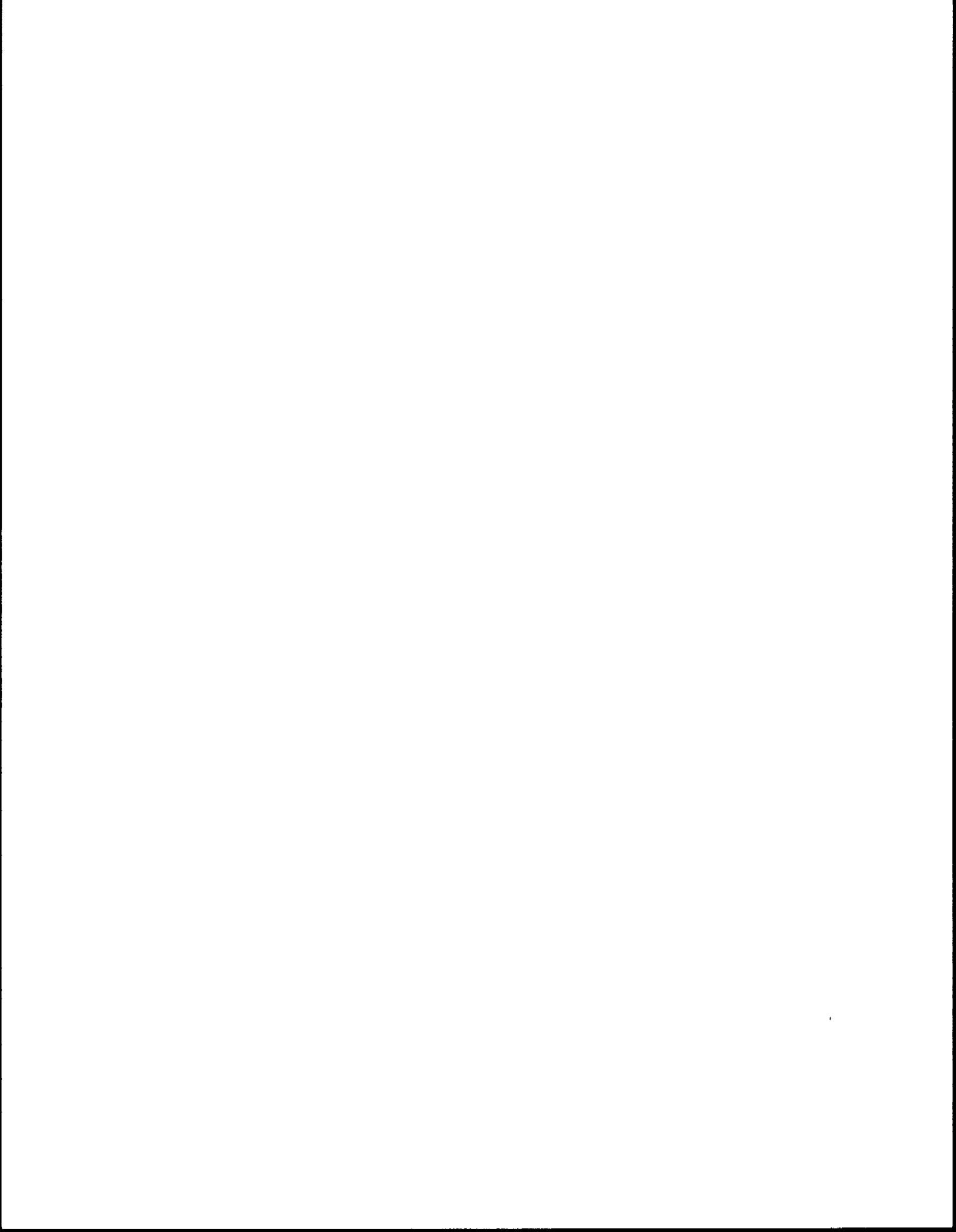
Principle of Operation

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

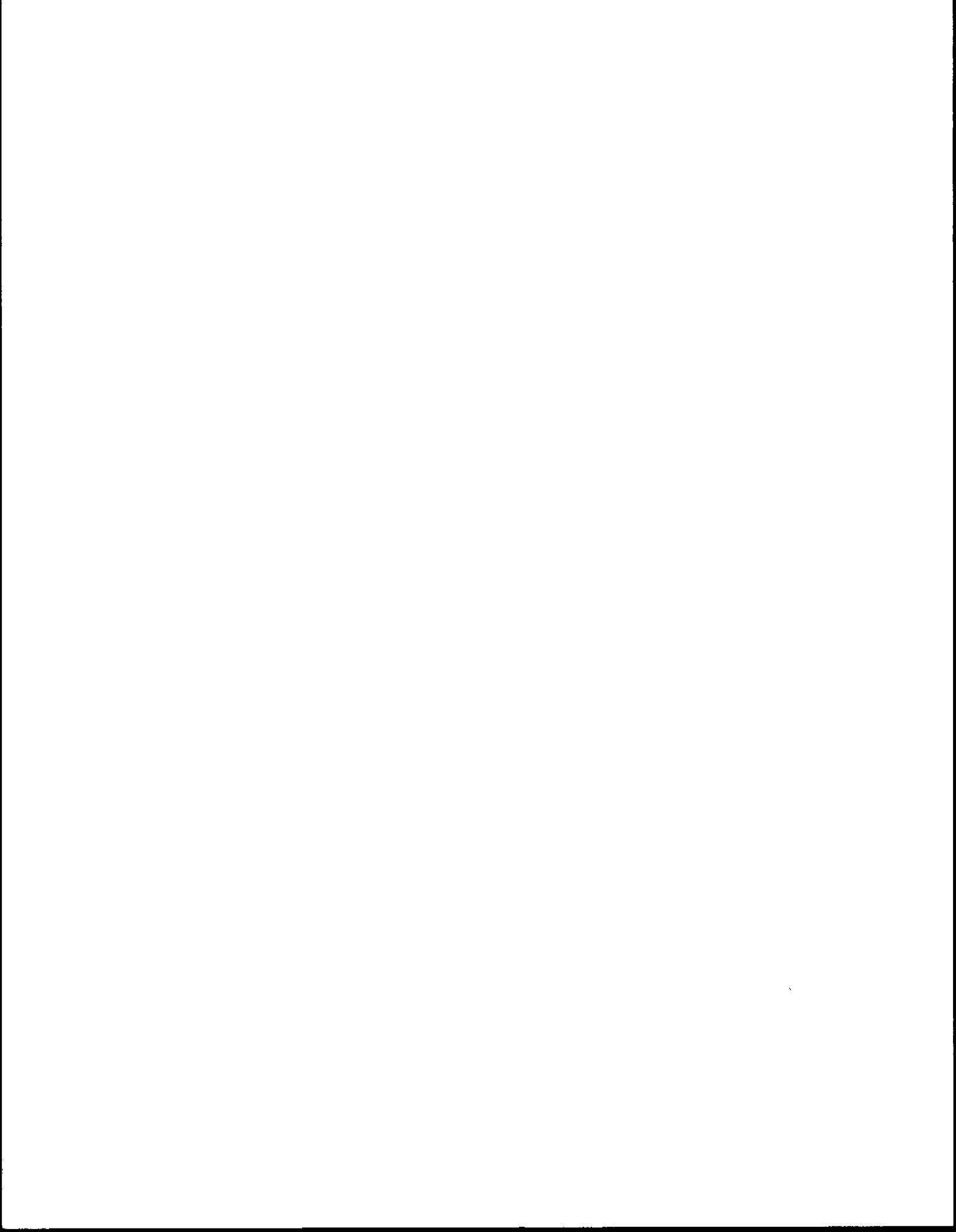
Spargers

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.

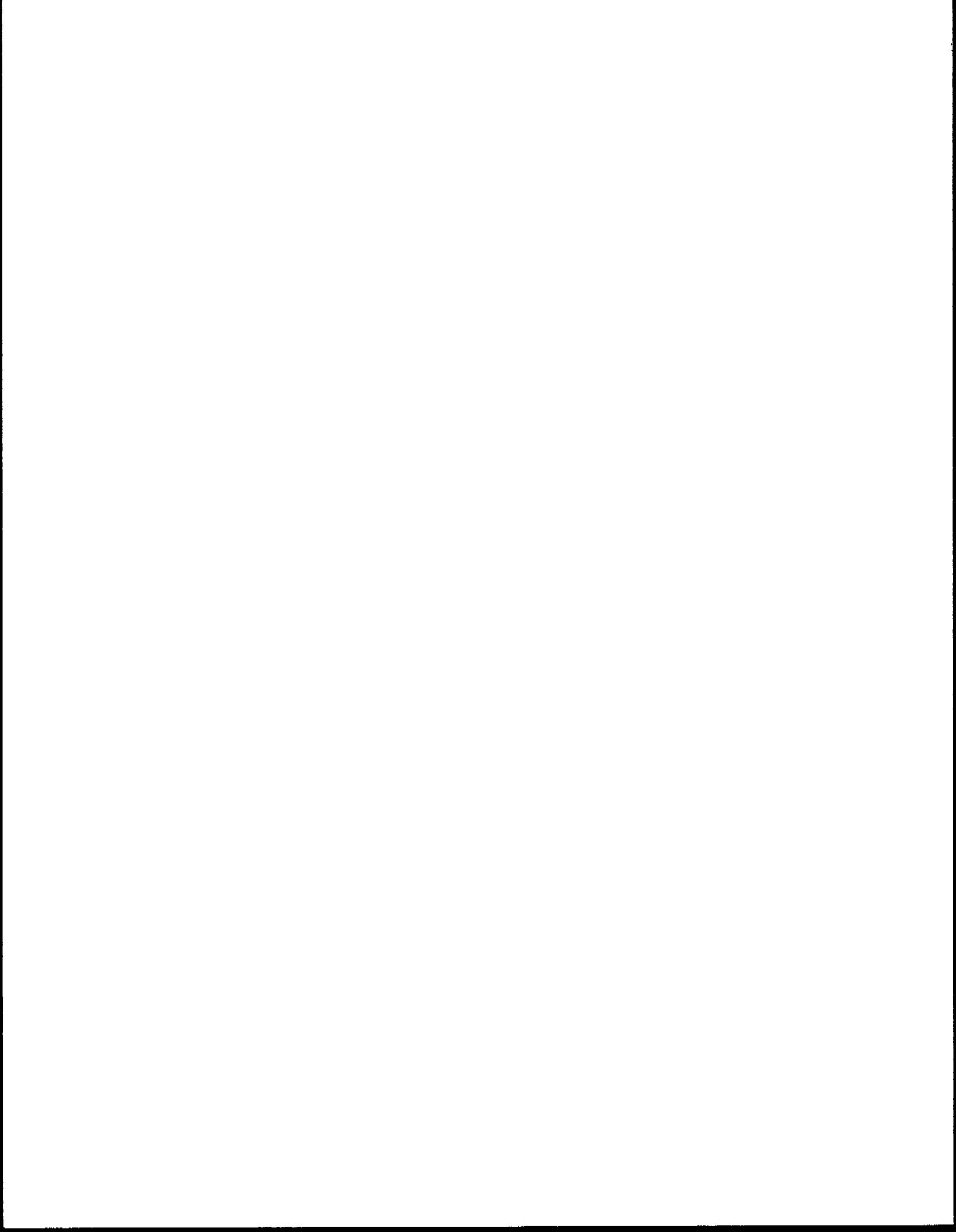
Programmable Ranges - Autosampler States

- 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

Power Consumption

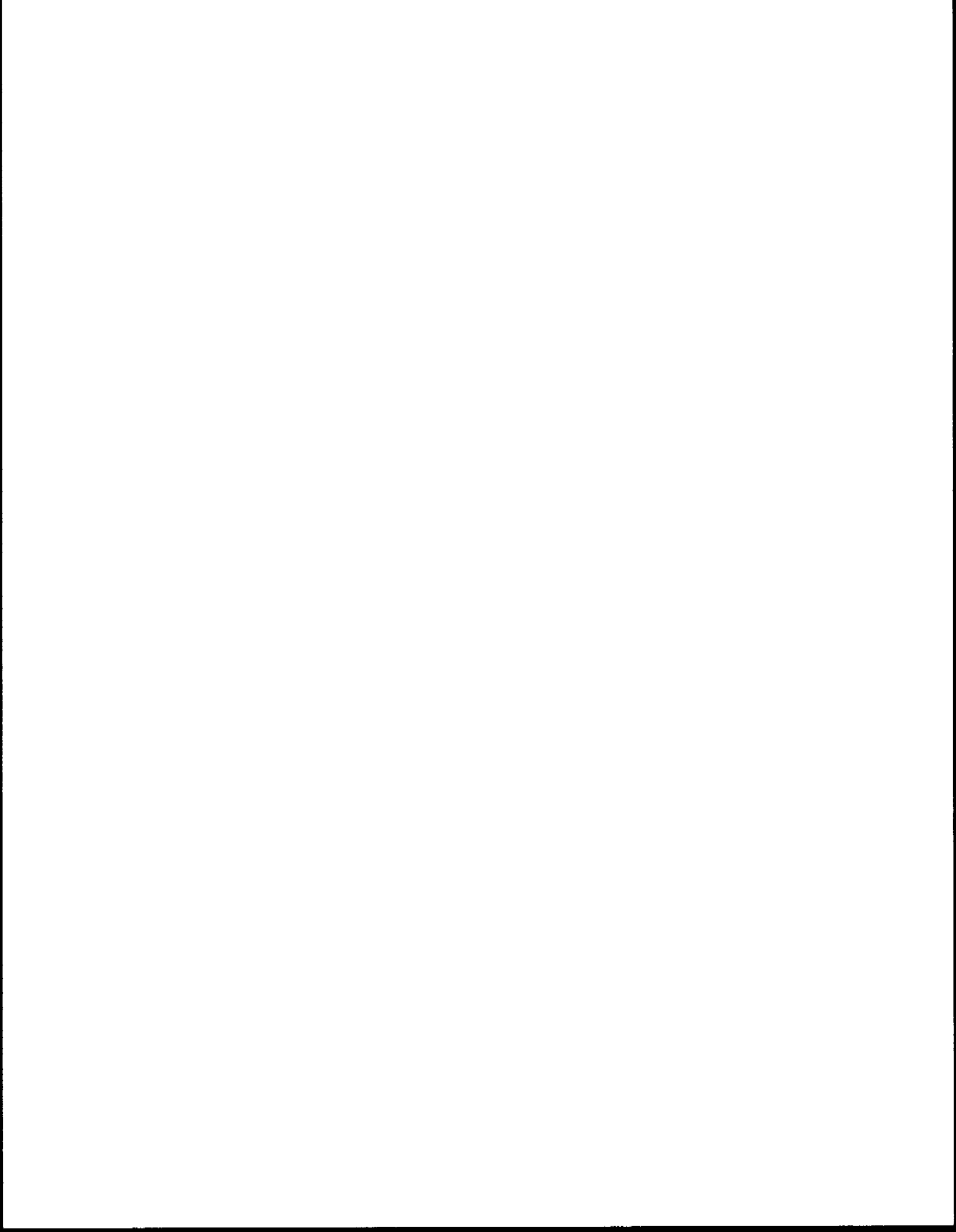
- 90 watts maximum

Line Voltage Noise Tolerance

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

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

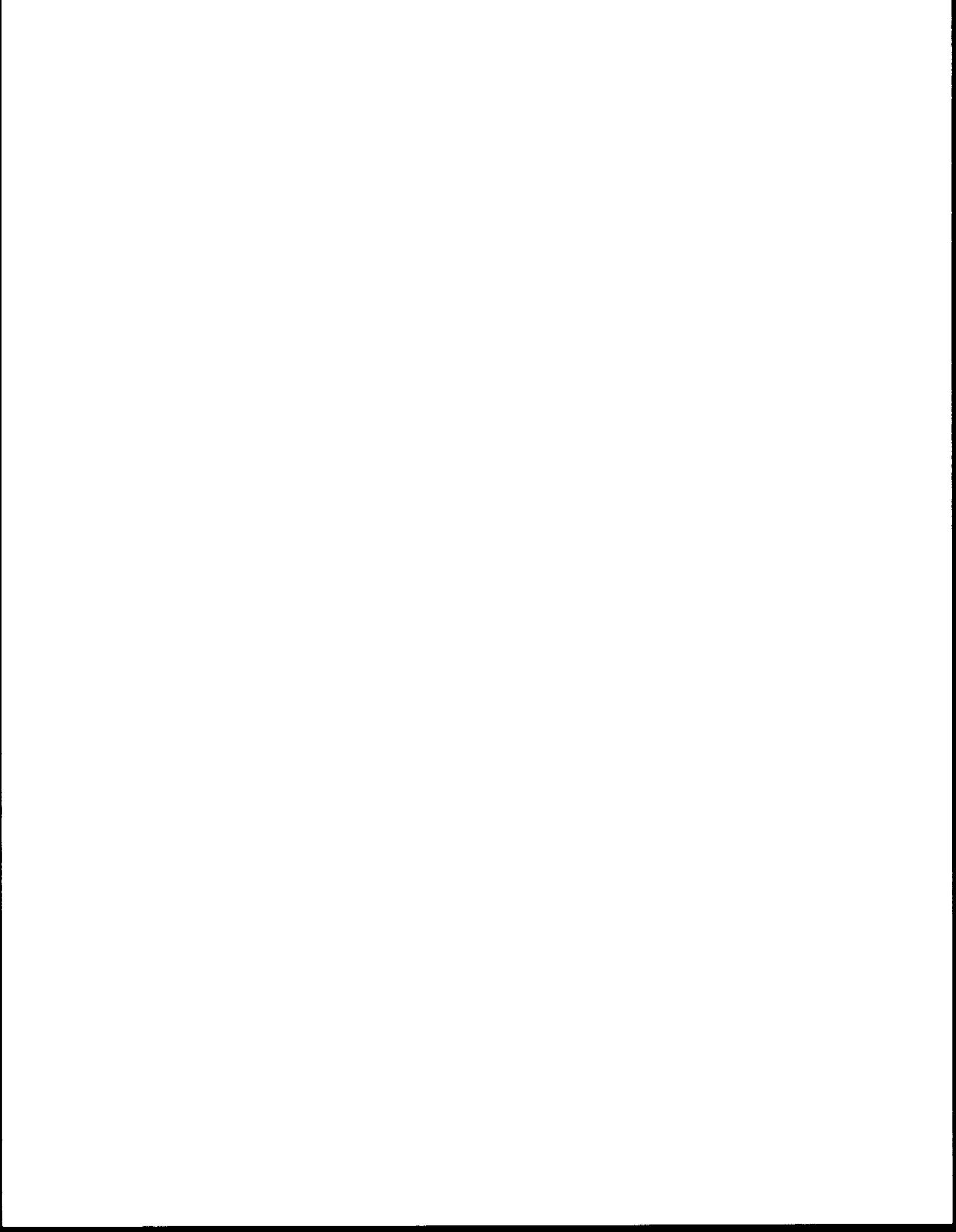
- Up to 100% relative humidity if connected to line voltage

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 μ l to 90 μ l in 10 μ l steps
- Injection Volume Precision: \pm 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

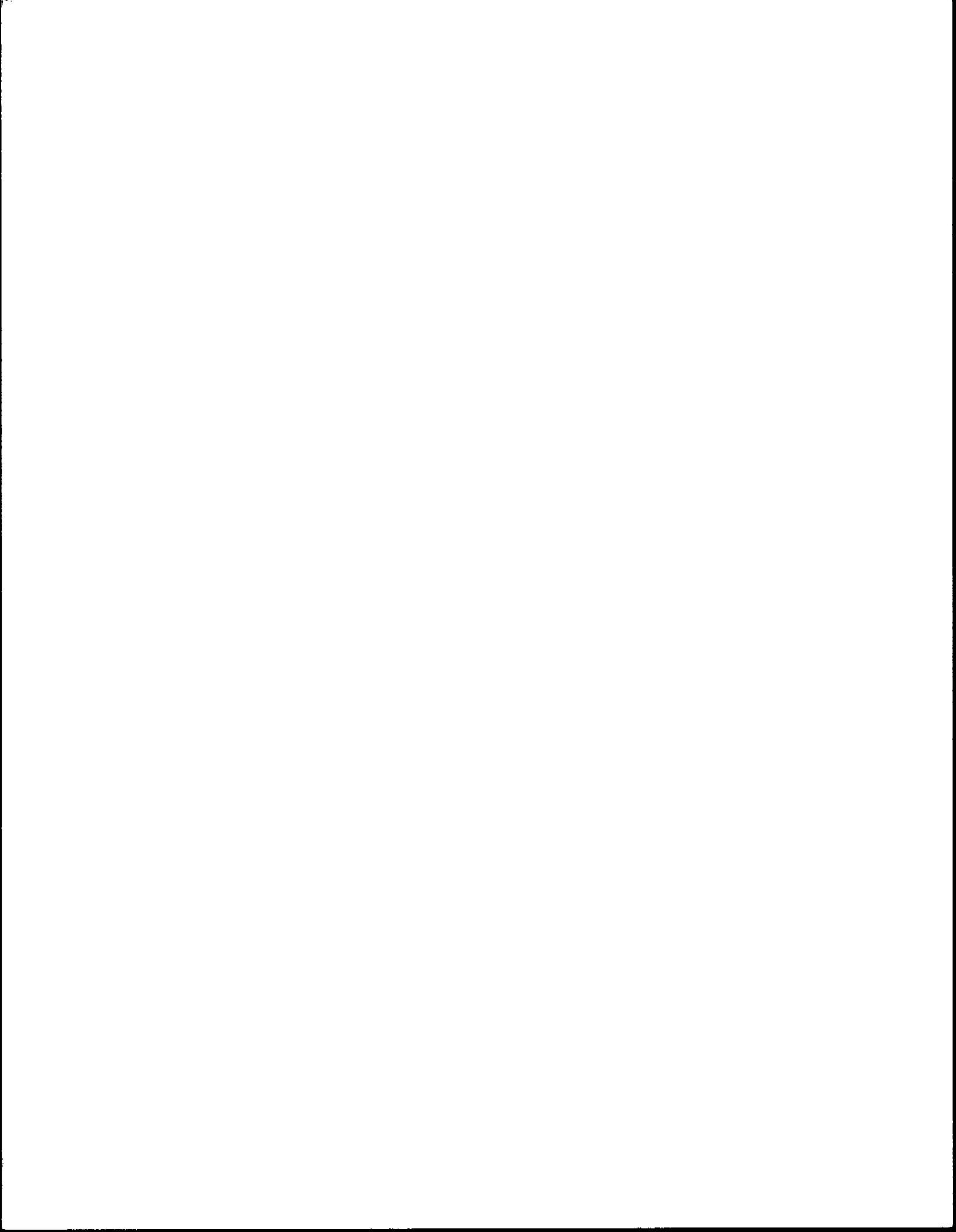
- 2.8 inches high
- 8 inches wide
- 11 inches deep
- 4 lbs

Electrical Requirements

- Power Supply: External, + 12 volts DC \pm 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 μ l to 90 μ l) 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)

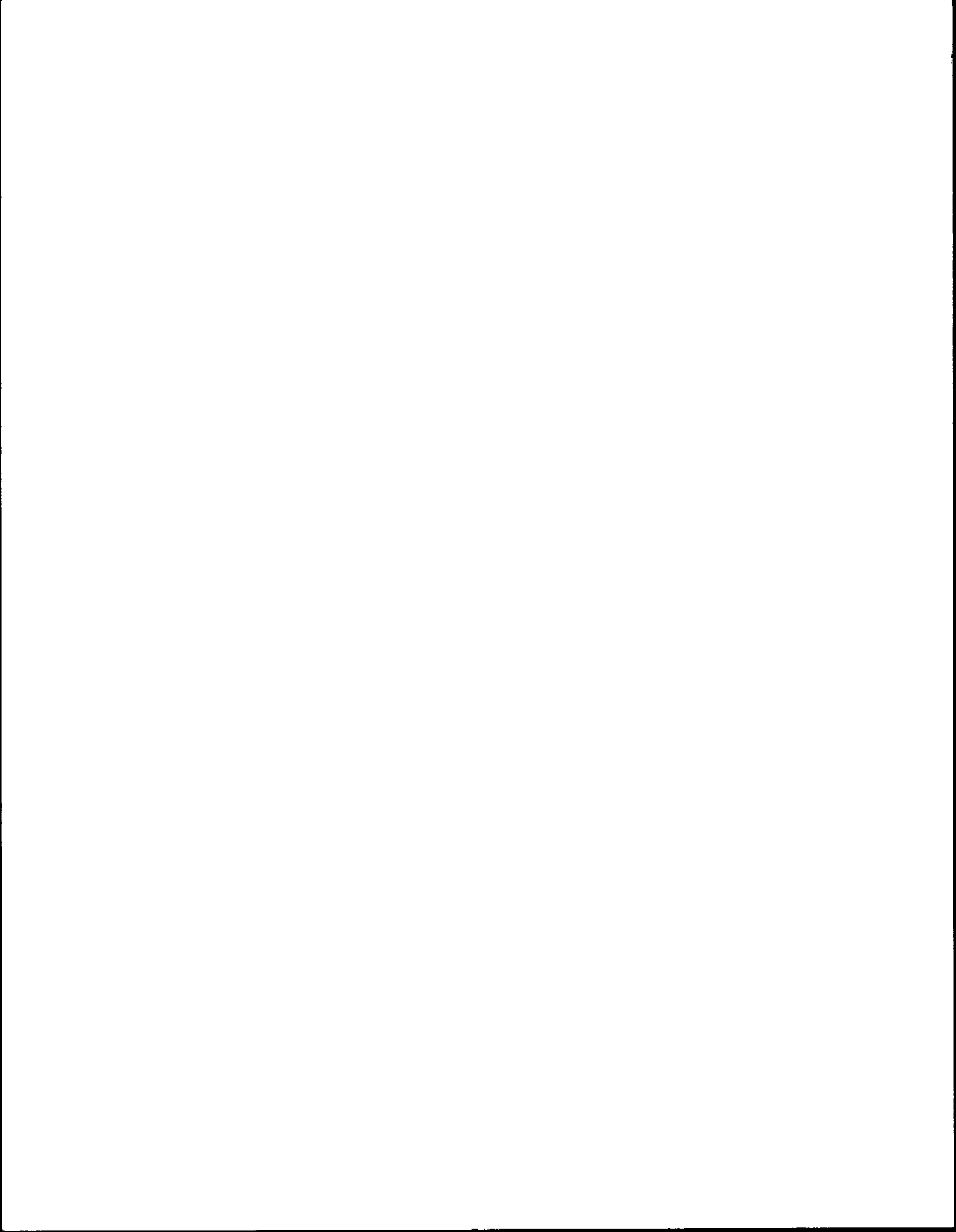
This kit includes two 5 ml syringes with 2-way valves for sample introduction. One of these syringes is included in the Startup Kit with the instrument, but stocking spares of all glass items is recommended.

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

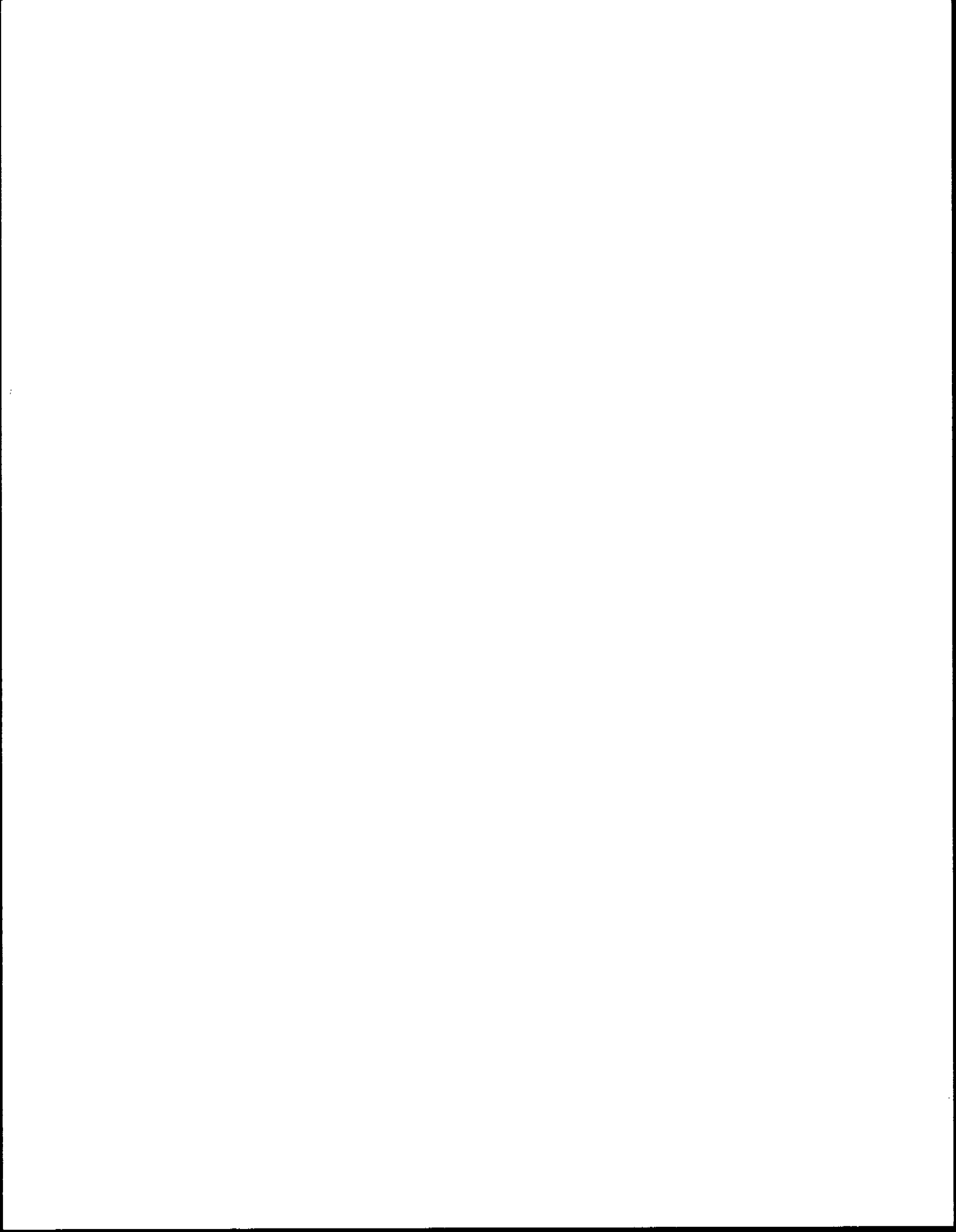
This kit is comprised of hardware necessary to interrupt the GC carrier gas flow immediately downstream of the flow controller (or its hydrocarbon scrubber if present), re-route this gas to the 4460A carrier gas inlet, and re-connect the leftover GC injector carrier line to the 4460A transfer line. The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

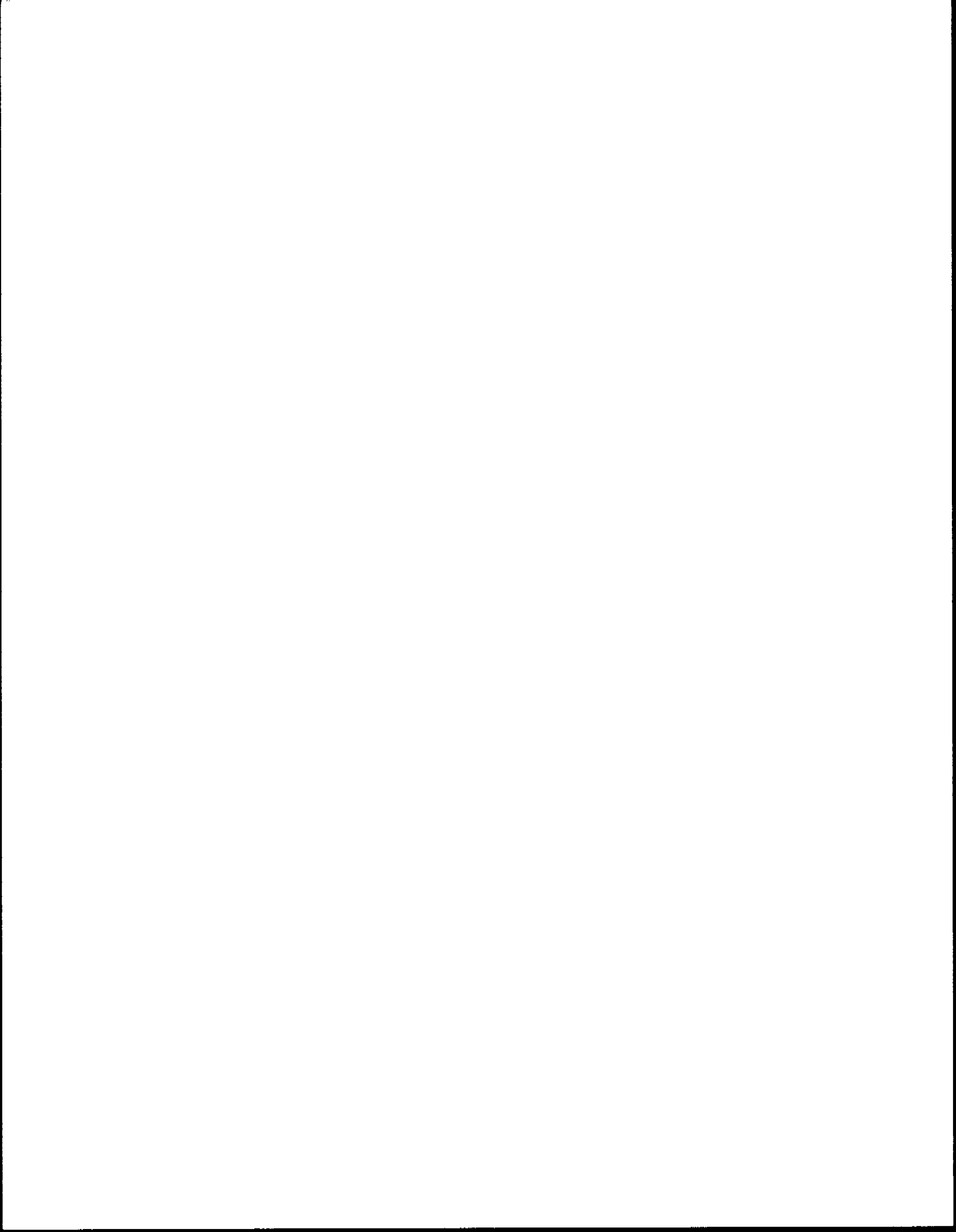
This manual valve is used to direct sample flow from a syringe into the **sample needle**, or from the **sample needle** to the **drain line**. The arrow on the valve handle points toward the path of flow. When the arrow is pointed to neither the **syringe port** nor the **drain line**, the **sample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

