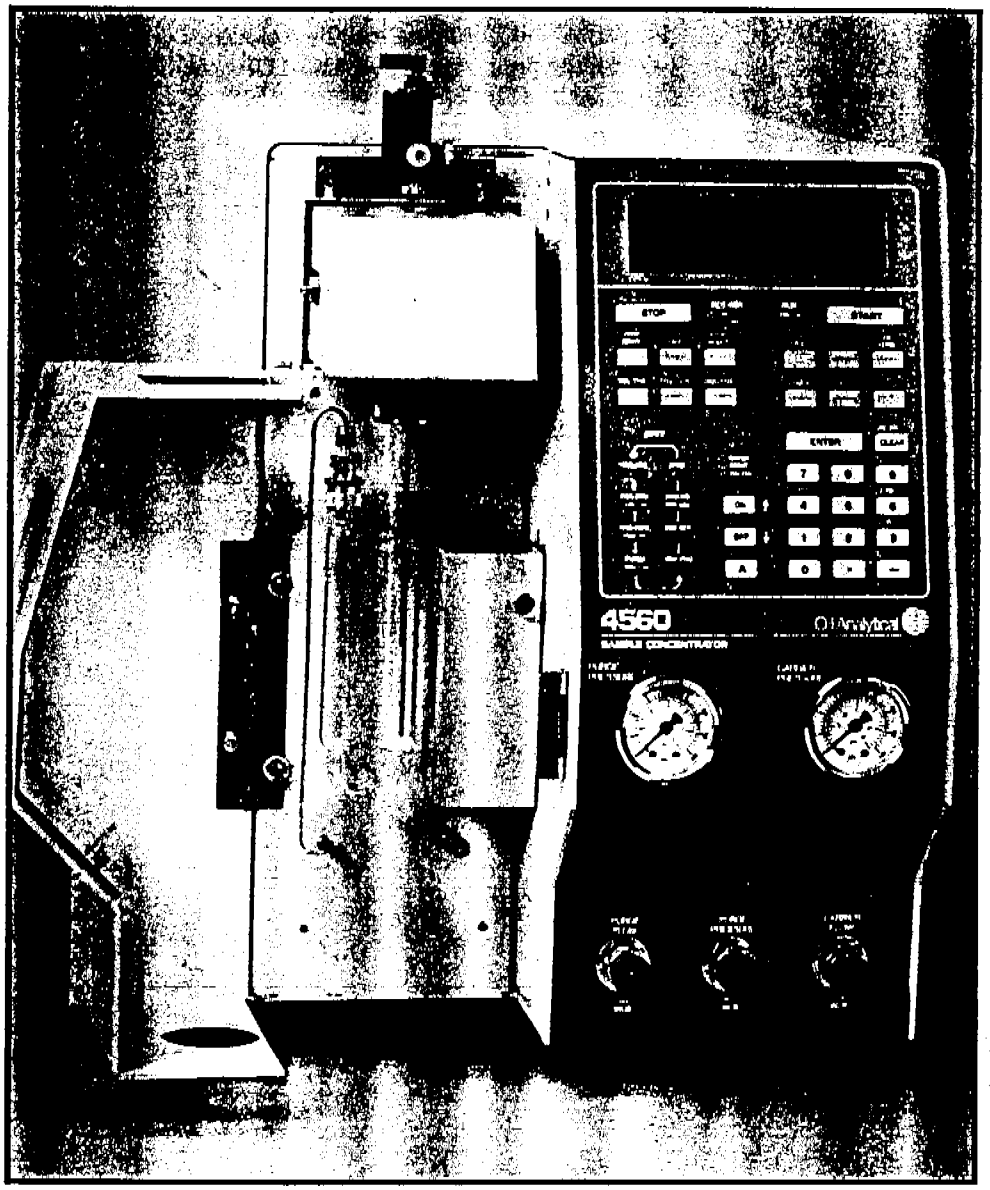
Model 4560 Sample Concentrator
Operator's Manual



O·I·Analytical

Graham Rd. at Wellborn Rd. · P.O. Box 2980 · College Station, Texas 77841-2980
Telephone (409) 690-1711 · FAX (409) 690-0440

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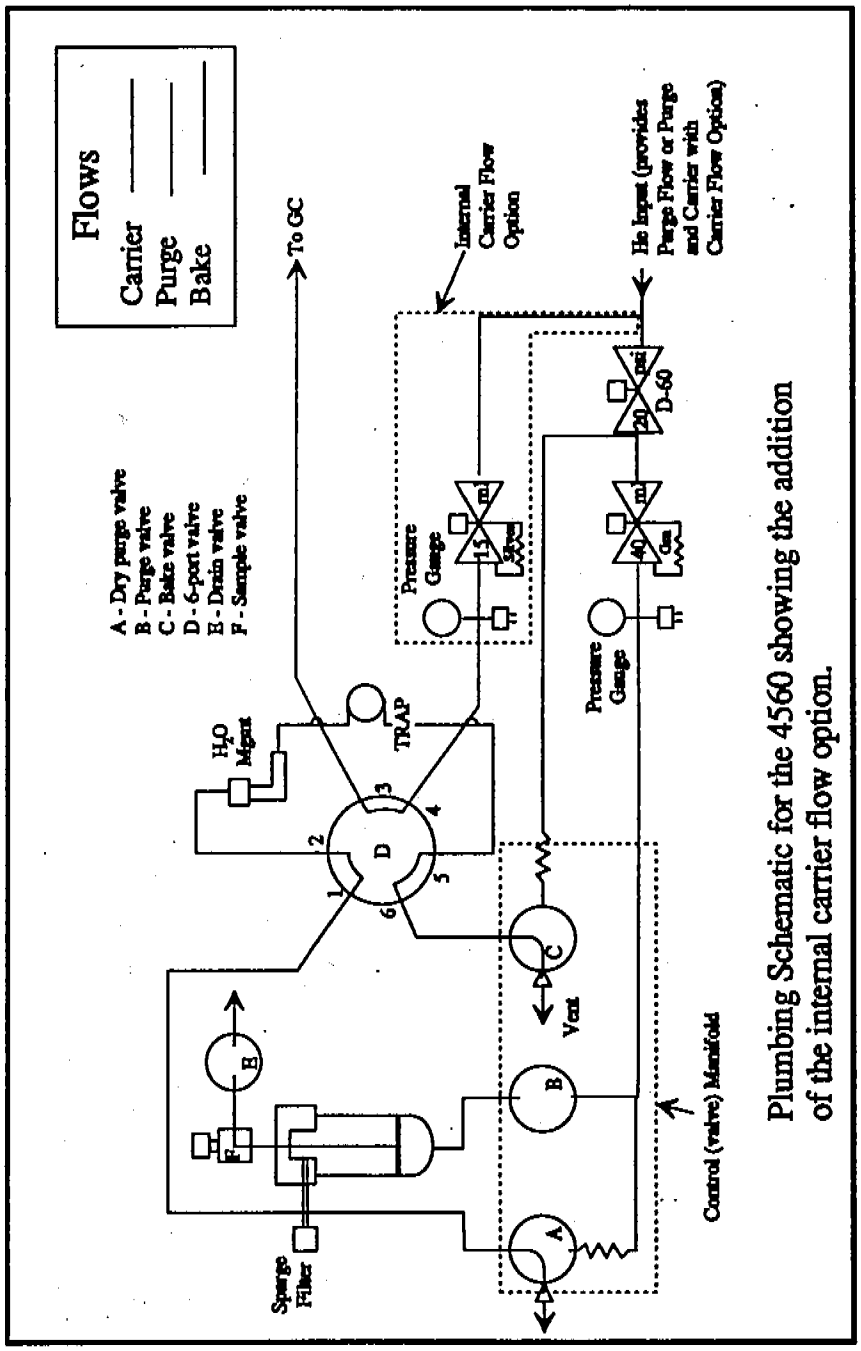
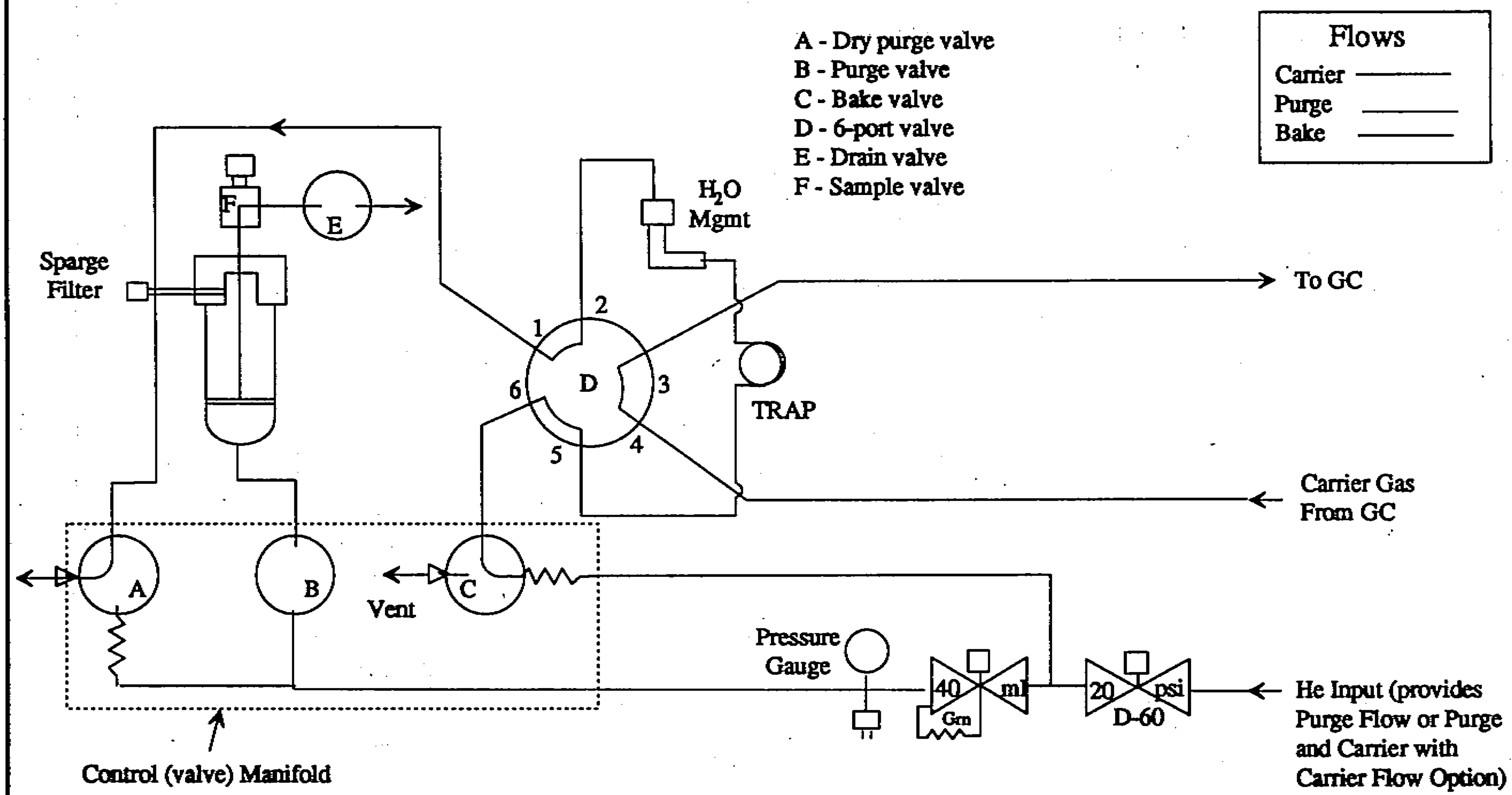
Chapter 6
Plumbing Schematics

This chapter presents schematic flow diagrams for each of the states, showing positions of valves and directions of flow



Plumbing Schematic for the 4560 showing the addition of the internal carrier flow option.


A - Dry purge valve
B - Purge valve
C - Bake valve
D - 6-port valve
E - Drain valve
F - Sample valve
Flows
Carrier
Purge
Bake
Sparge Filter
F
E
H2O Mgmt
1
2
3
4
5
6
D
TRAP
To GC
Carrier Gas From GC
A
B
Vent
C
Pressure Gauge
40
ml
Gm
20
psi
D-60
He Input (provides Purge Flow or Purge and Carrier with Carrier Flow Option)
Control (valve) Manifold

STANDBY

Table of Contents

Chapter 1

Chapter 2

Chapter 3

Chapter 4

Chapter 5

Chapter 6

Chapter 7

Chapter 1
Introduction

The Model 4560 is a new generation Purge-and-Trap Sample Concentrator used to collect and transfer organic compounds to a GC or GC/MS for analysis. It is specifically designed to purge volatiles from water and soil for concentration onto a sorbent trap in strict compliance with EPA protocol. The sample transfer line connects directly to the column or through an optional Low-Dead-Volume Interface™ to the GC column in an analytical system. The 4560 features tactile keypad entry and a vacuum-fluorescent display to enhance ease of use.

Principle of Operation

Liquid, solid, or gaseous (from a solid support) samples containing volatile organic compounds are sparged (with a regulated flow of inert gas for a fixed period of time. Analytes stripped from the sample (or transferred from an upstream multisampling product) are concentrated on a cool sorbent trap specific to the application. The trap is then rapidly heated and, with a valve change, the analytes are desorbed as a "plug" under reversed flow of carrier gas onto the GC column. Virtually all water transferred from the sample matrix to the trap during sparging remains in the concentrator. The water is then baked out to vent, reducing interference with subsequent reconcentration, separation, or detection of the analytes.

Design Features

- Operator interface is extremely friendly and simple to use. Vacuum-fluorescent display is easy to read.
- Optional Infra-Sparge™ Sample Heater provides temperature feedback from within sample and safety shut-off.
- Built-in Cyclone Water Management™ minimizes the amount of water transferred to the analytical column.
- Provides for 5 and 25 ml frit and needle spargers, and disposable tube spargers, using needle of same length and 18 mm mount.
- Provides means for handling sample foaming and spillover with removable particle filter in sample inlet.
- Provides controlled temperature of each heated zone.
- Processor-controlled. Fully interactive via RS-232 communication port.
- Basic operational sequence controlled by key entry; inter-instrument communication may be achieved through simple contact closures.

- Up to 14 sets of run-settings can be saved, loaded as files, and even sequenced during a multisampler run.
- Rapid trap heating (900° C/min) and cooling (~250° C/min) optimizes chromatography and run times.
- All lines that touch the sample are inert and impermeable to gases.
- Incorporates power-up self-test, and diagnostics messages.
- Optional Flow Controller allows all gases to be provided by and monitored through the Sample Concentrator.
- Incorporates BCD inputs for multisampler position monitoring.

Specifications

General Specifications

Dimensions
- 14.5" H x 10.25" W x 14.2" D
- Footprint - 145 in²

Weight
- 31 lbs

Programmable Time Range
- 0 - 999.99 min for all time parameters

Trap
- 0.125" O.D. x 0.105" I.D.
- Coil shape
- Stainless steel
- Direct resistive heating

Valve
- Electrically actuated
- 6-port, 60° rotation
- Removable rotor

Programmable Temperature Ranges
- Trap: ambient to 300° C in PURGE, DESORB, and BAKE
- Sample Transfer Line: ambient to 200° C
- Valve Oven: ambient to 300° C
- Sample Inlet: ambient to 200° C
- External Heater: ambient to 300° C
- Optional Sample Heater: ambient to 100° C

Transfer Line
- Nickel
- 48" standard
- 60" optional
- Fused-silica lined stainless steel optional

Column Compatibility
- 0.20 - 0.53 mm with Cryo-Focusing Module™
- 0.32 - 0.53 mm (or packed) without Cryo-Focusing Module™

Standard Glassware
- 5 ml frit sparger (18 mm neck)

Optional Glassware
- 5 ml Needle Sparger (18 mm neck)
- 25 ml Needle Sparger (18 mm neck)
- 25 ml Frit Sparger (18 mm neck)
- 20 ml Disposable Test Tubes (18 mm neck)

Electronic Control
- 80188 microprocessor
- 128K ROM
- 32K RAM
- 14 programmable methods with a battery backup
- STE Bus connector
- Tactile, elastomeric keypad
- 2 x 20 dot matrix, alpha-numeric, vacuum-fluorescent display

Performance Specifications

Trap
- 900° C/min heating rate (25° C to 180° C in <11 sec)
- ≈250° C/min cooling rate (180° C to 25° C in approximately 40 sec)

Optional Sample Heater
- Up to 30° C/min sample heating rate
- ±1° C temperature accuracy for 5 or 25 ml samples

Temperature Accuracy
- ±1° C for *all* heated zones

Temperature Stability
- ±1° C for *all* heated zones

Water Management
- <.3 µl of water transferred to column at sparge temperatures to 85° C
- Operates at *ambient* temperature
- Water removal at level equivalent to condensation at 4.8° C
- Adds no time to purge-and-trap cycle

Communications

Output Signals
- 2 sec contact closure at PURGE READY, START DESORB, and START BAKE

Input Signals
- 50 ms contact closure at PURGE READY to PURGE and DESORB READY to DESORB

Communication Interface
- Standard RS-232C (bi-directional)
- 9600 baud rate
- Optional Purge and Trap Total Interface (PATTI®II) software package
- O•I•NET™ network interface for inter-instrument communication

Requirements

Gas Requirements

- 99.999% He or N_2 purge gas

Power Requirements

- Standard unit - 115 VAC (±10%)/50/60 Hz (800 VA max)
- Available unit for - 230 VAC (±10%)/50/60 Hz operation

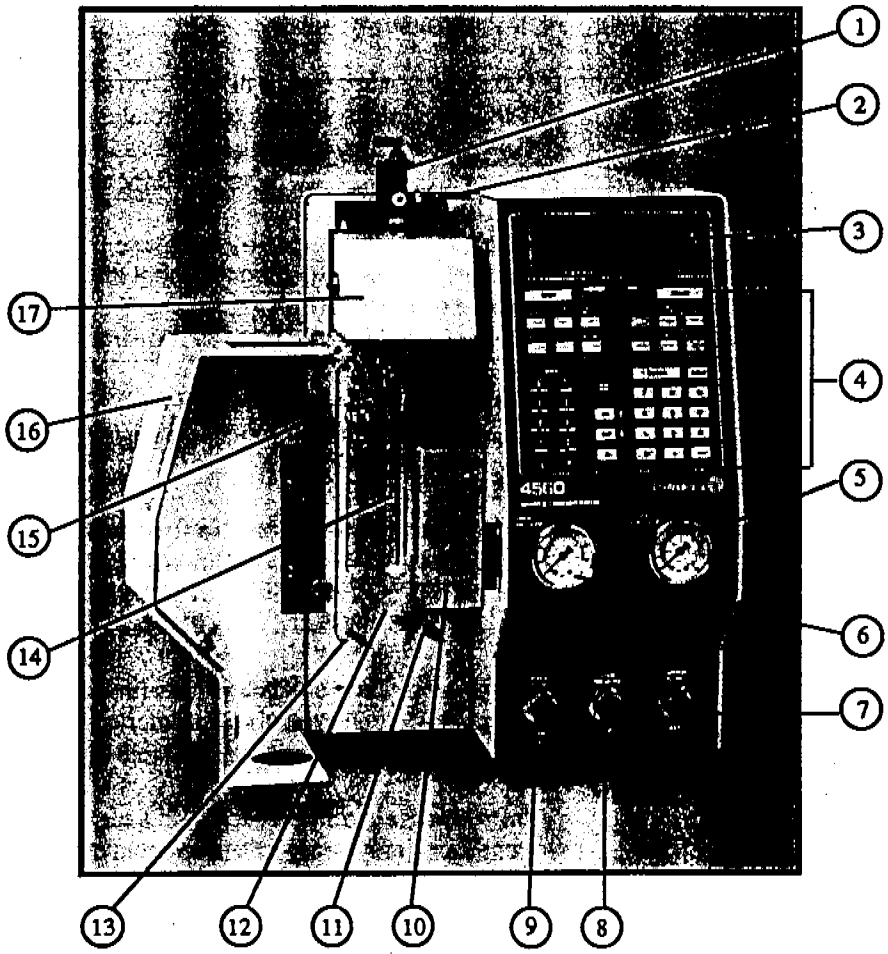
Major Options

- Internal Carrier Flow Control (OI Analytical Part #227934)
- PATTI®II Windows™ Control Software (OI Analytical Part #232389)
- Infra-Sparge™ Sample Heater (OI Analytical Part #225649)
- Cryo-Focusing Module™ (OI Analytical Part #210120)

Chapter 2
Description of Components

Exterior Description of Components

Front Panel



1. Sample Valve
2. Drain Line
3. Display Screen
4. Keypad
5. Carrier Pressure Gauge (optional)
6. Purge Pressure Gauge
7. Carrier Flow Control Knob (optional)
8. Purge Pressure Regulator Knob
9. Purge Flow Control Knob
10. Infra-Sparge Sample Heater (optional)
11. Purge Flow Outlet
12. Purge Vessel
13. Purge Gas Supply Outlet
14. Purge/Drain Needle
15. Frit Sparge Line
16. Purge Vessel Door
17. Sparge Mount Cover

***Fig. 2.1** Model 4560 Front View*

Sample Valve is used to manually syringe-inject (into Luer-lock injection port fitting) a *water* sample into the Purge Vessel. This Valve is also used to connect the Drain Line if frit sparging, or the Purge Gas Line if needle sparging.

Drain Line is used to drain *water* sample automatically during DESORB. This silicone Line runs through a pinch Drain Valve in the interior right bay of the 4560, which opens when it is time to drain the sample. The Drain Line leads to the Sample Drain Port on the rear panel of the unit and is connected to the Sample Valve by a knurled 10/32" nut and stainless steel ferrule.

Display Screen is a vacuum-fluorescent display used to view selected settings/parameters.

Keypad (Tactile elastomeric) is used to enter settings and select parameters displayed on the Display Screen.

Carrier Pressure Gauge displays carrier (head) pressure from 0-60 psi (0-400 kPa). This Gauge, along with the corresponding Carrier Flow Controller, is optional. The Gauge allows easy routing of all necessary gases (carrier and purge) to the Sample Concentrator, eliminating the need for a GC injector in some cases.

Purge Pressure Gauge displays purge pressure from 0-60 psi (0-400 kPa). Purge pressure is often used to troubleshoot the unit, relative to expected pressures in each state (e.g., DESORB). This reading is dependent on Column length, Column diameter, carrier flow rate, and oven temperature.

Carrier Flow Control Knob adjusts Column carrier flow from the Sample Concentrator. The Controller is supplied with a 0-15 ml/min restrictor (for most capillary column applications).

Purge Pressure Regulator Knob adjusts incoming purge gas pressure, supplying purge gas to the Purge Flow Controller. Turning this Knob clockwise increases the purge gas pressure supplied to the Purge Flow Controller. The required incoming purge gas pressure setting is 20 psi in PURGE READY.

Purge Flow Control Knob adjusts flow of gas supplied by the Purge Gas Pressure Regulator. Turning this Knob counter-clockwise increases the amount of purge gas passed through the sample each minute (usually 30-40 ml/min).

Infra-Sparge™ Sample Heater is a high-intensity bulb used to heat water or soil samples. It has a removable aluminum cover with built-in filter to block visible light. It is controlled and monitored through temperature feedback from inside the sample (see *Internal Sample Thermocouple*). The Sample Heater is optional.

> **Lamp Contacts** (not shown) provide power to light Lamp as needed to maintain sample set temperature.
>
> **Lamp** (not shown) is an expendable component easily replaced as necessary.
>
> **Safety Shut-off Switch** (not shown) interrupts power to Lamp when Purge Vessel Door is opened.

Purge Flow Outlet (10/32" barbed fitting) is the vent for the purge gas flow. The location of the fitting allows for easy measurement of the purge flow for adjustment or monitoring, without removing the cover from the unit.

Purge Vessel provides a reservoir for purging of the sample. Vessels are available in a 5 ml or 25 ml size, in either a frit style (usually for clean-water samples) or needle-sparge style (for particulated or soil/solid waste samples).

Purge Gas Supply Outlet is an exterior fitting used to connect the frit-style or needle-style Purge Gas Line to appropriate Purge Vessel.

Purge/Drain Needle supplies purge gas directly into sample when needle sparging. It acts as drain pathway when frit sparging.

Frit Sparge Line connects to the Purge Gas Supply Fitting and runs to Purge Inlet Arm of frit-style sparger. This Line supplies purge gas to the frit when frit sparging.

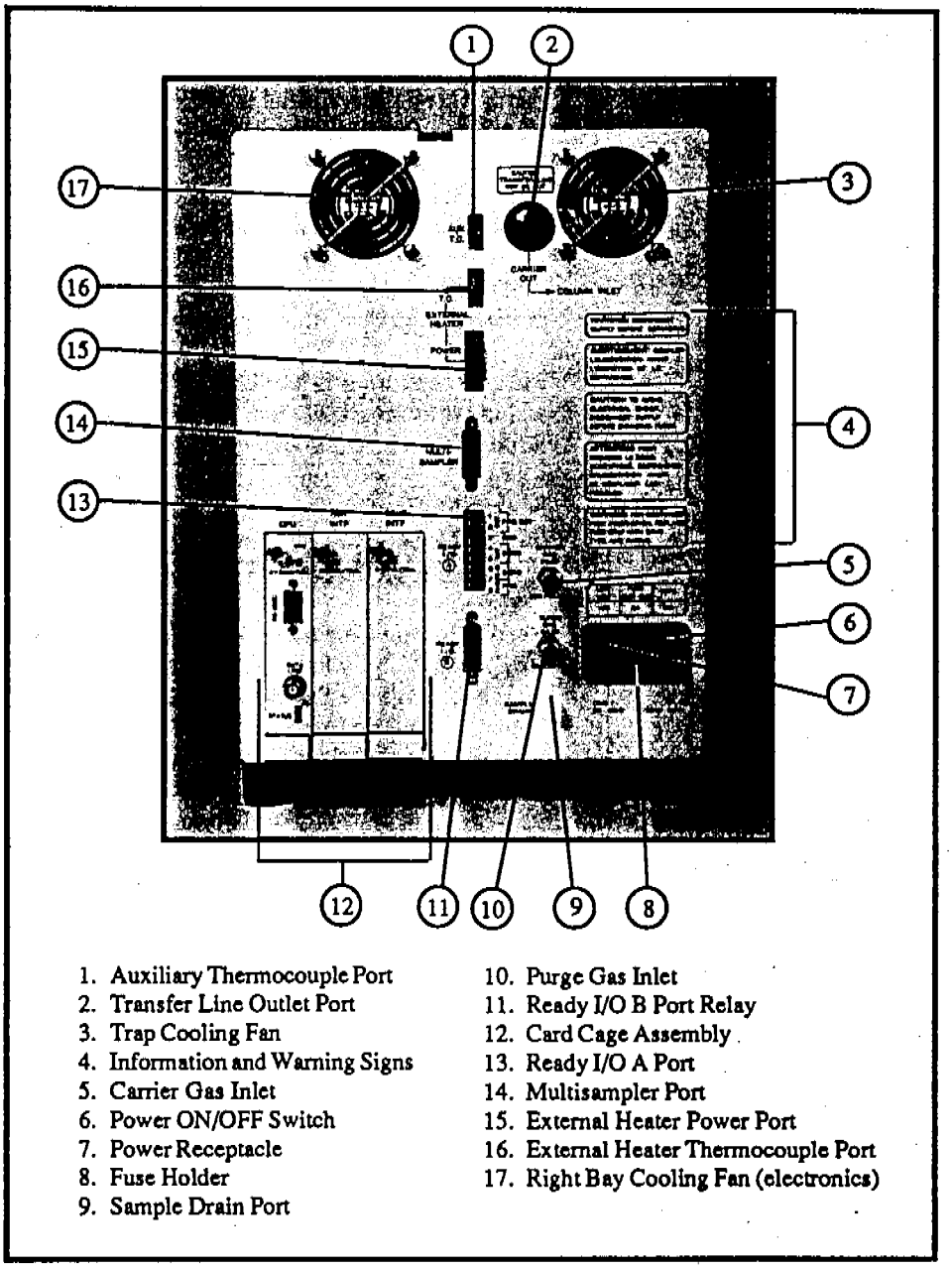
Purge Vessel Door is provided to protect glassware from accidental breakage, for safety, and to aid in sample heating. It allows the operator to visually monitor the sample through the window in the door.

Sparge Mount Cover provides a mount for the Purge Vessel and insulation for the Sample Mount Heater. This Cover slides forward to expose the Sample Inlet and forward Valve Oven area.

Needle Sparge Line (not shown) connects to the Purge Gas Supply Outlet and runs to the Sample Valve when needle sparging. This Line supplies purge gas to a needle-style sparger through the Sample Purge Needle.

Internal Sample Thermocouple (not shown) provides direct sample temperature feedback to the Sample Heater for temperature monitoring and control of water or soil samples.

Rear Panel



1. Auxiliary Thermocouple Port
2. Transfer Line Outlet Port
3. Trap Cooling Fan
4. Information and Warning Signs
5. Carrier Gas Inlet
6. Power ON/OFF Switch
7. Power Receptacle
8. Fuse Holder
9. Sample Drain Port
10. Purge Gas Inlet
11. Ready I/O B Port Relay
12. Card Cage Assembly
13. Ready I/O A Port
14. Multisampler Port
15. External Heater Power Port
16. External Heater Thermocouple Port
17. Right Bay Cooling Fan (electronics)

Fig. 2.2 Model 4560 Rear View

Auxiliary Thermocouple Port (Type K thermocouple) is used to monitor the temperature of a selected heated zone. It usually monitors the oven temperature to signal a "GC Ready" to the Concentrator when no "GC Ready" contact closure is available from the GC.

Transfer Line (not shown) is a heated line used to carry volatiles desorbed from the Trap to the GC. Temperature is operator-selectable.

Transfer Line Outlet Port provides port for the Transfer Line to exit the Concentrator and allows for mobility of the Line.

Trap Cooling Fan cools the Concentrator's Trap, as required, during the purge-and-trap cycle.

Information and Warning Signs warn the operator of potential hazards associated with improper use of the instrument and inform the operator of voltage requirements.

Carrier Gas Inlet is used to connect the 1/16" Carrier Gas Supply Line from an external flow controller (e.g., the GC Injector Flow Controller). Supply carrier gas at 250 psi maximum pressure. This Inlet should be plugged when the Carrier Flow Control Option is present on the 4560.

Power ON/OFF Switch is the power control switch. Turning ON the unit is accompanied by a power-up self test.

Power Receptacle is used to connect the 4560 to an appropriate power source via a cable provided in the Start-Up Kit.

Fuse Holder contains the main fuse used to protect the 4560 from a short circuit condition.

Sample Drain Port is a fitting that connects to the silicone Drain Line provided in Start-Up Kit. The operator should route this Drain Line to a waste receptacle.

Purge Gas Inlet is used to connect the 1/8" Purge Gas Line from the secondary regulator at 30-250 psi. **(The Purge Gas Inlet also supplies carrier gas to the GC when the Internal Carrier Flow Control Option is present on the 4560.)**

Ready I/O B Port (15-pin D-sub connector) provides hard-wire communication interface from the 4560 to any GC System providing and accepting the required contact closures. This requires a standard OI Analytical interface cable for your specific brand of GC.

Card Cage Assembly contains slots for the insertion or removal of the CPU Card, an Auxiliary Interface Card, and an optional OI Analytical Cryo-Focusing Module™ (CFM™) Card. CPU Card allows for connection of an RS-232 cable, has a port used for data linking, and contains status LEDs used for CPU Card troubleshooting.

Ready I/O A Port (8-pin Phoenix connector) provides hard-wire communication interface from the 4560 to any GC System providing and accepting the required contact closures. This port may be used when no GC specific interface cable is available.

Multisampler Port (15-pin D-sub connector) provides communication line connection from the 4560 to any OI Analytical Multisampler (see *Communication Cabling*, pp. 21-22).

External Heater Power Port (3-pin Amp connector) provides power to external heated zone, as necessary, to maintain temperature setpoint (see **External Heater Thermocouple Port**).

External Heater Thermocouple Port (Type K thermocouple) is used to monitor an external heated zone (e.g., the MPM-16 Valve Oven and Heated Lines).

Right Bay Cooling Fan cools the electronic components and transformer in the right bay of the 4560 when the temperature exceeds 30° C.

Left Side-View

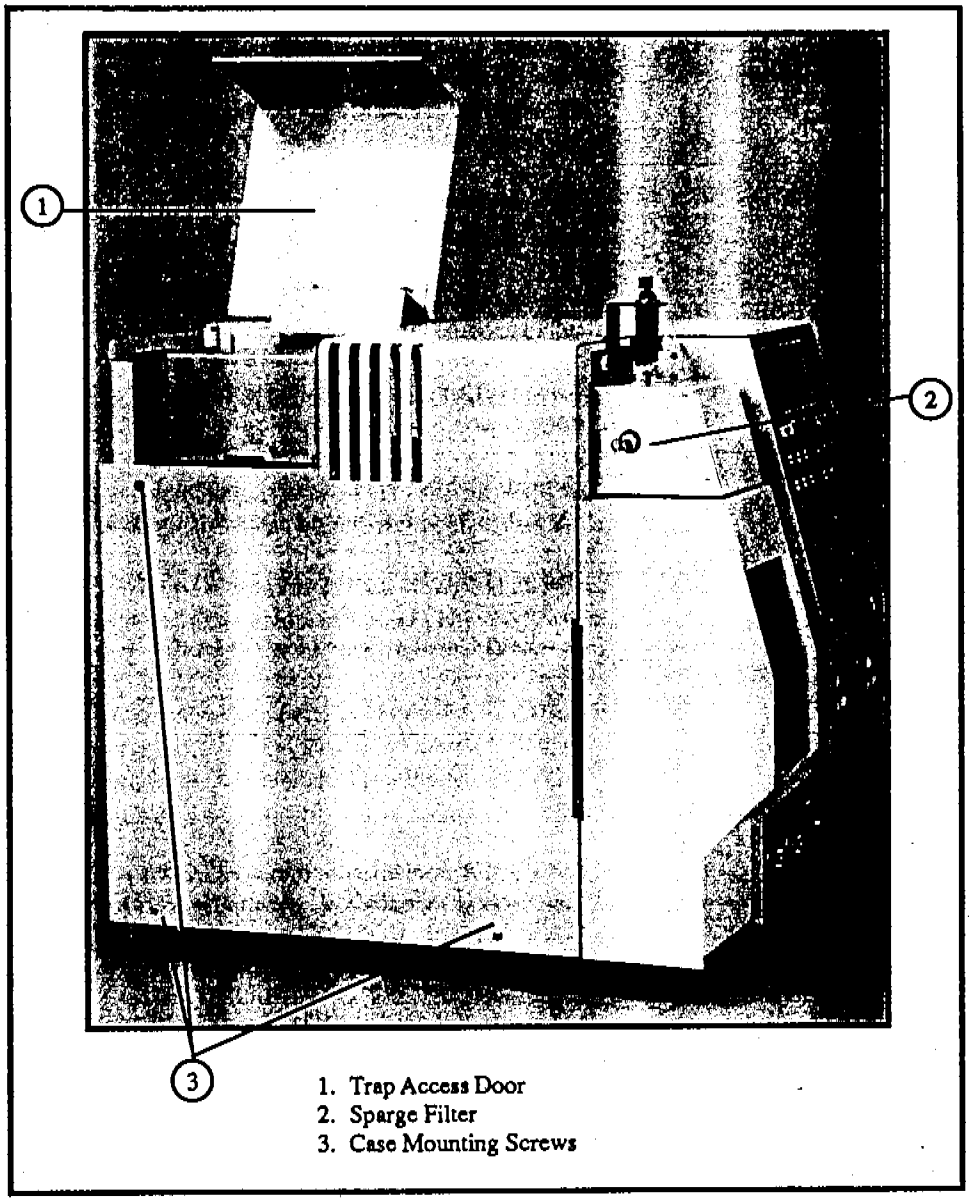


Fig. 2.3 Model 4560 Left Side

Trap Access Door allows access to the Concentrator's Trap without requiring the removal of the instrument cover. When this door is opened, a Safety Shut-off Switch cuts power to the Trap.

Sparge Filter protects the Trap from foaming samples often encountered in purge-and-trap analysis. It may be replaced as an expendable part or cleaned via the procedure outlined in this manual. If an OI Analytical MPM-16 Multisampler is present, the Sparge Filter is removed and the port is used to connect the Heated Transfer Line from the MPM-16.

Case Mounting Screws secure the cover to the main chassis of the 4560.

Right Side-View

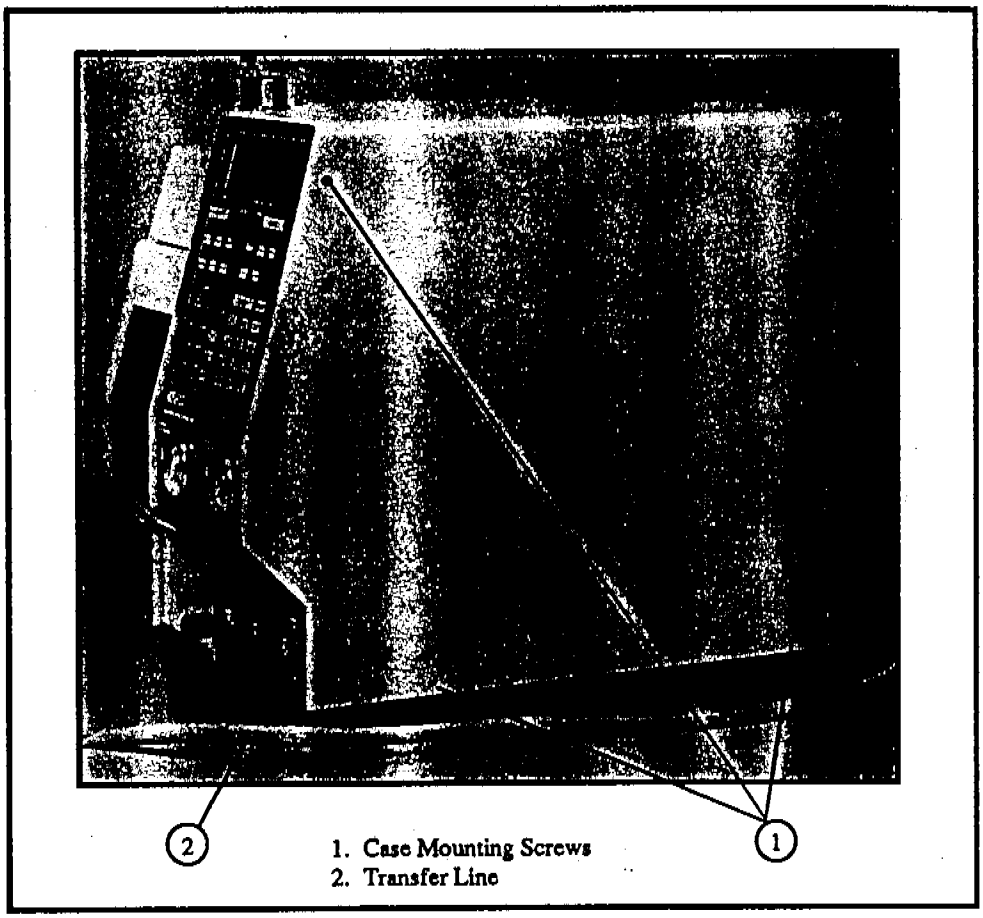


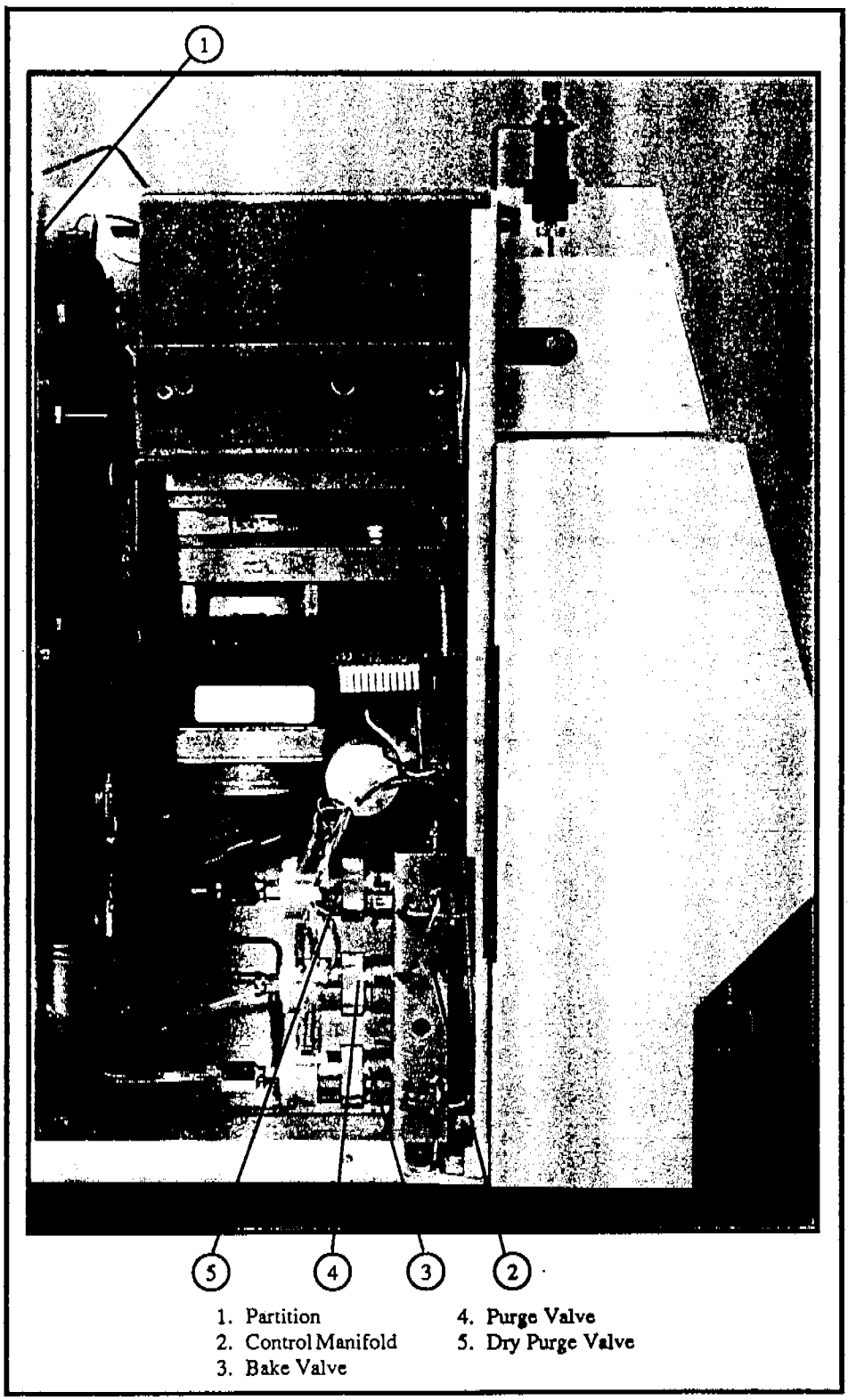
Fig. 2.4 Model 4560 Right Side

Case Mounting Screws secure the cover to the main chassis of the 4560.

Transfer Line (see Rear Panel Description).

Interior Description of Components

Left Bay



1. Partition
2. Control Manifold
3. Bake Valve
4. Purge Valve
5. Dry Purge Valve

Fig. 2.5a Model 4560 Left Bay

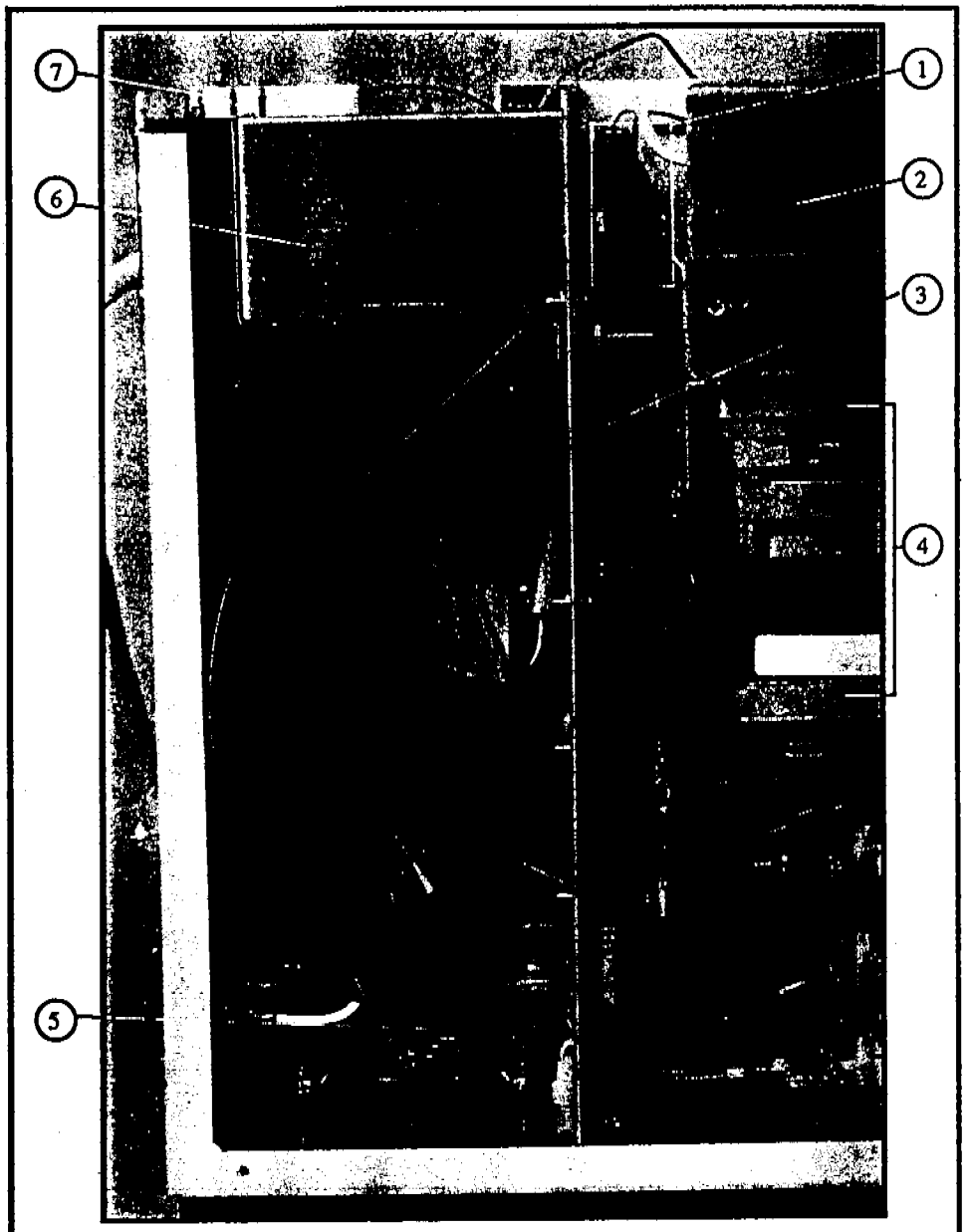
Partition provides mounting surface for Water Management Cooling Fan and Trap Assembly.

Control Manifold provides heat for and directs flow to the Purge, Dry Purge, and Bake Valves. Temperature for this manifold is preset and cannot be changed, only monitored.

Bake Valve is a 3-way brass/nickel solenoid that is the final vent for purge gas flow. A Check Valve on the end of the solenoid prevents atmospheric air from entering the system.

Dry Purge Valve is a 3-way brass/nickel solenoid that is used to re-direct flow to bypass the sparger for DRY PURGE. It acts as the water management vent during BAKE. A Check Valve on the end of the solenoid prevents atmospheric air from entering the system.

Purge Valve is a 3-way brass/nickel solenoid that opens to allow purge flow to the Purge Vessel.



1. Water Management Assembly
2. 6-port Valve Oven Assembly
3. Water Management Cooling Fan
4. 6-port Valve Actuator Assembly
5. Left Bay Cooling Fan (Intake Fan)
6. Trap Cover
7. Trap Safety Shut-off Switch

Fig. 2.5b Model 4560 Left Bay

Water Management Assembly is a proprietary assembly that manages and vents water purged onto the trap during DESORB and BAKE.

6-port Valve Oven Assembly is a heated housing for the 6-port Valve and all associated lines running to and from the Valve.

Valve Oven Cover insulates the 6-port Valve Oven Assembly. This Cover is removed by lifting vertically.

Water Management Cooling Fan cools the Water Management Assembly (as required) during the purge-and-trap cycle.

6-port Valve Actuator Assembly electronically actuates 6-port Valve 60° in DESORB and back for next sample run.

Left Bay Cooling Fan keeps internal temperature of left bay at proper temperature when instrument cover is present.

Gas Flow Splitter splits incoming Purge Gas Line to provide gas to Carrier Gas Flow Controller, when the option is present.

Trap Cover insulates the Trap. This cover is removed by pressing in on both sides and lifting vertically. Trap Access Door allows the removal of the Trap Cover without removing the instrument cover.

Trap Safety Shut-off Switch interrupts power to the Trap when the Trap Access Door is opened. Trap will cool when Trap Access Door is opened.

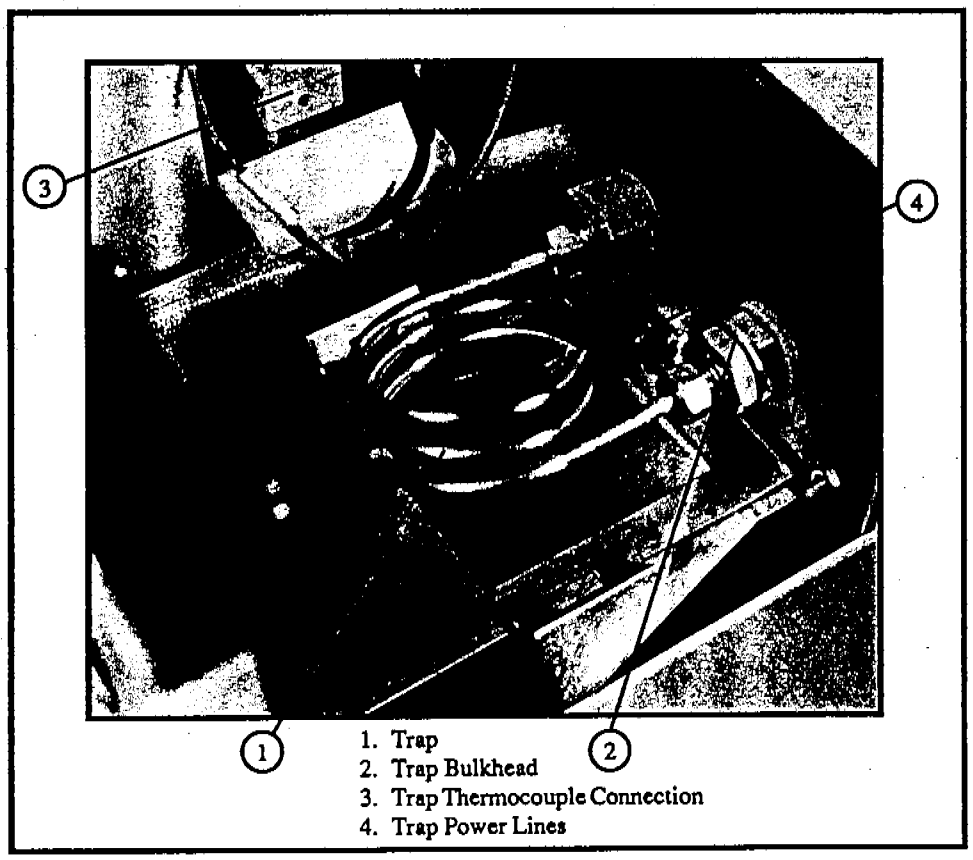


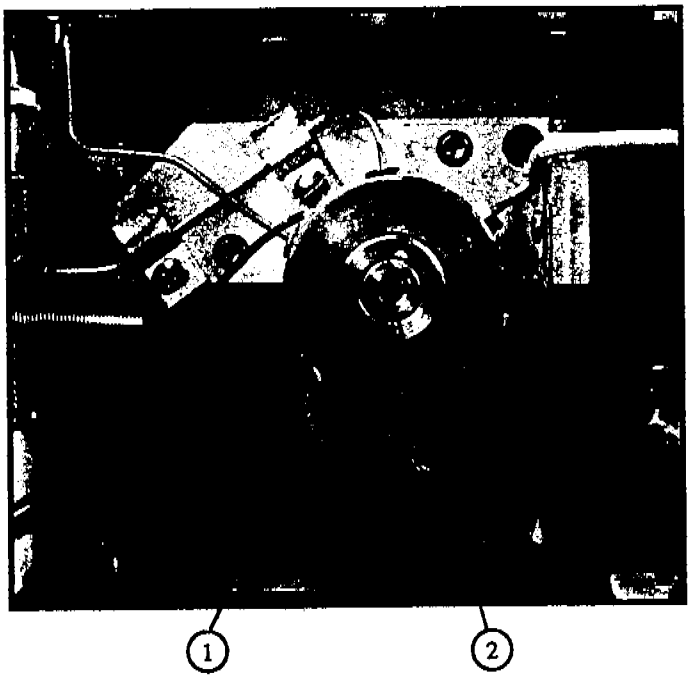
Fig. 2.6 Model 4560 Trap

Trap is used to trap volatile components purged from the sample.

Trap Bulkhead (gold-plated) provides inert connection of the lines routed from the 6-port Valve to the Trap.

Trap Thermocouple Connection provides Trap temperature feedback to the AC/IO board on the 4560.

Trap Power Lines (not shown) provide current directly to the Trap Bulkhead to heat the Trap.



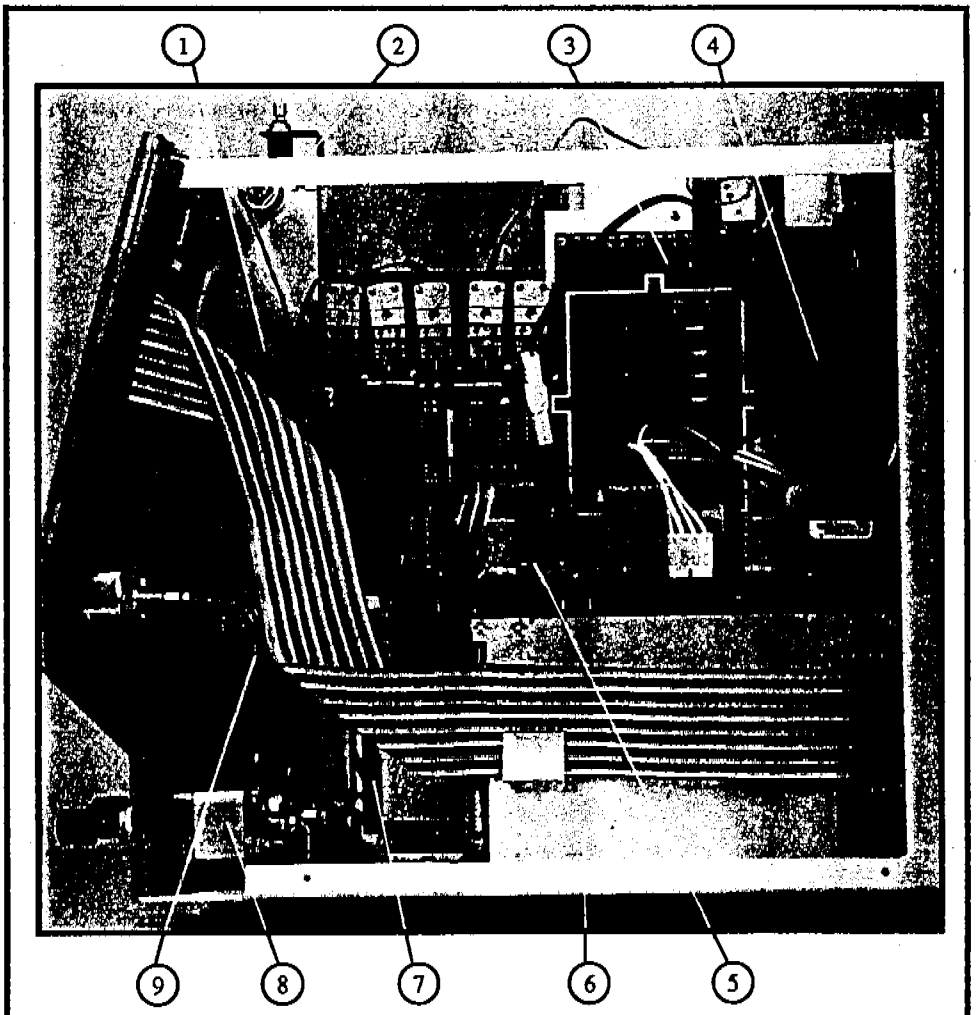
1. 6-port Valve Auxiliary Heater Ring
2. 6-port Valve

Fig. 2.7 Model 4560 6-port Valve Auxiliary Heater Ring

6-port Valve Auxiliary Heater Ring heats (operator-selectable temperature) upper portion of 6-port Valve.

6-port Valve rotates 60° as required to place Trap in-line with analytical Column.

Right Bay



1. Sample Heater Safety Shut-off Switch
2. Drain Valve (partially hidden)
3. AC/IO Board
4. Transformer
5. Power Supply Board (horizontal)
6. Card Cage Assembly
7. Back-Plane Board
8. Carrier Flow Controller (optional)
9. Sample Heater Power Supply Assembly

Fig. 2.8 Model 4560 Right Bay

Display Boards provide electronics for vacuum-fluorescent display and Keypad.

Sample Heater Safety Shut-off Switch (see Front Panel description).

Drain Valve (see Drain Line description).

AC/IO Board is the large PC board located on the side panel that separates the electronics and the mechanical components of the 4560. This Board has analog conditioning circuitry and connectors for the thermocouple inputs. The switching elements for controlling the heaters and fuses are located under the blue cover that is attached to the PC board. The connectors for the heaters and the AC power are located on the back side of this Board. These connectors are accessible from the mechanical side of the 4560 through a hole in the side panel. The external I/O signals for interfacing to other analytical equipment are on this Board and are located on the back panel of the 4560. There are LEDs located on this Board that indicate the status of the Valves,

Fans, and heated zones. Protection is incorporated for the detection of open thermocouple inputs. If the overtemp LED (D100) is ON, power to all heated zones has been shut off. This will occur if any of the thermocouple inputs are open. Unused thermocouple inputs should be terminated by a dummy thermocouple.

Transformer provides power to the Trap.

Power Supply Board is attached to the top of the card cage assembly. Its function is to supply DC power to run the electronics. It supplies the following DC voltages: +12 V, -12 V, +15 V, - 15 V, and +5 V.

Card Cage Assembly (see Rear Panel description).

Back-Plane Board is a common board for all boards residing in Card Cage.

Carrier Flow Controller (see Front Panel description).

Sample Heater Power Supply Assembly supplies power to Sample Heater described in **Front Panel** description.

Purge Flow Controller (not visible in picture).

Purge Pressure Regulator (not visible in picture).

CPU Card is located in the first slot of the Card Cage. It is the controller for all functions of the 4560. It is used to measure and control the temperature of all the heated zones in the 4560. This card also communicates with the operator by controlling the Keypad and display on the front panel. The RS-232 port is accessed through the 9-pin connector on the back of the Card. This is the same CPU Card (except for the EPROM Chips) used in the OI Analytical 5220 ELCD.

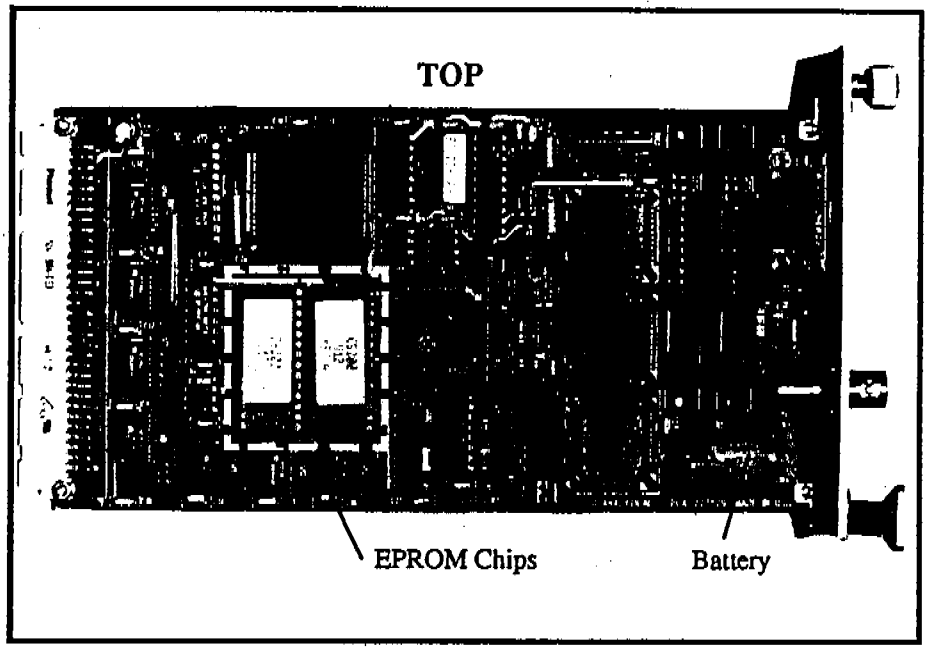


Fig. 2.9 CPU Card

Firmware can be upgraded for the unit by replacing the EPROM Memory Chips (OI Analytical Part #228106) on the CPU Card. To replace EPROM Memory Chips, follow these instructions.

- Turn 4560 OFF.
- Remove the CPU Card from the unit.
- To remove old EPROM Memory Chips (U17 and U18), gently pry up with an extractor or a small flat-head screwdriver if an extractor is not available.
- When inserting the pins of the Chips into the appropriate holes in the CPU Card, make sure the pins are not bent.
- **Pay particular attention to the position of the notches at the tops of the chips. The notches must "point to" the top of the CPU Card. Orient the Chips as shown in Fig. 2.9.** Insert the Chip marked "U17" into the area on the CPU Card marked "U17." Similarly, insert the Chip marked "U18" into the area on the CPU Card marked "U18." **The chips must be inserted completely and correctly.**

The CPU Card's Lithium Battery should function properly for several years. The 4560 is designed to retain current settings after the unit is turned OFF. If settings are not retained, the Battery may be dead. Perform the following steps to replace the Battery.

- Turn 4560 OFF.
- Remove the CPU Card from the unit.
- To remove old Battery, gently pry up with a small flat-head screwdriver.
- **Orient the Battery as shown in Fig. 2.9.** The "+" lead (as marked on the Battery's side) should be inserted into the socket also marked with a "+." After proper Battery orientation is confirmed, insert both leads into their corresponding sockets.

Fuses are used to protect the electronics from failing if a short circuit condition occurs with one of the heated zones. The main fuse is located in the Power Entry Module on the back of the unit where the AC cord is connected to the unit. To remove the fuse, the cord must be removed so the cover on the Power Entry Module can be removed using a screwdriver. The fuse holder will slide out and the fuse can be replaced. Fuses are also located under the blue cover on the AC/IO Board. These can be replaced once the cover has been removed. If a fuse is blown, the condition that caused the fuse to open should be discovered before it is replaced. The fuses are labeled so that the heated zone that caused the failure can be inspected before the fuse is replaced.

Chapter 3
Installation

This chapter describes the installation of the Sample Concentrator. The installer should read through this information, gather the materials needed, then proceed in a stepwise fashion through the instructions beginning with **Unpacking/Positioning the Instruments.**

Materials Needed

The following is a list of materials not provided with the basic Sample Concentrator but which are necessary for its installation.

Purge Gas Source: A source of purge gas (99.999% pure He or N_2), regulated to a pressure between 30 and 250 psi (207-1724 kPa) is needed for connection to the Purge Gas Inlet (50 psi is recommended). External scrubbing of gas is highly recommended. Solvent-rinsed, baked copper tubing should be plumbed by the operator to within 5 ft of the 4560 (see Gas Hookup Lines).

Carrier Gas Source: A source of carrier gas must be provided to the Flow Controller of the GC interfaced to the Sample Concentrator. The purity and pressure requirements of this source should be as specified by the manufacturer of the GC or GC detectors being used. For the Sample Concentrator, 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 the purge gas. When the Internal Carrier Flow Option is present on the 4560, the flow is split internally to each pressure regulator (purge and carrier).

Gas Hookup Lines: A clean stainless steel or copper 1/8" (3.2 mm) O.D. Gas Hookup Line is required to connect the purge gas source to the Purge Gas Inlet of the Sample Concentrator (5 ft of copper tubing is provided).

GC Interface Kit: A Kit for interfacing the Sample Concentrator to the GC is needed for connection of the GC Carrier Gas Outlet to the Sample Concentrator Carrier Gas Inlet and for the connection of the Sample Concentrator Transfer Line to the injection port of the GC. An OI Analytical Low-Dead-Volume Interface™ Kit is available for most popular GCs. The LDV™ Kit contains necessary hardware for interfacing to the GC as well as remote start (handshake) cabling.

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.

Unpacking/Positioning the Instrument

- Remove the Startup Kit and the Sample Concentrator from the shipping carton. Save all packing material until proper operation of the Sample Concentrator is verified.

- Place the Sample Concentrator next to the GC on the side nearest the GC injection port. The space under the Sample Concentrator should be kept clear of obstructions for proper air flow during operation. Placing the 4560 on a hot surface may inhibit proper trap cooling and affect results.

Sparger (Purge Vessel) Installation

The 4560 arrives configured with a 5 ml frit sparger (see Fig. 3.1). If you choose to convert to a 25 ml frit sparger, see instructions for changing the sparger in Chapter 5. If you purchased a Needle Sparge Hardware Kit (OI Analytical Part #227397) and wish to needle sparge, follow instructions in the next section.

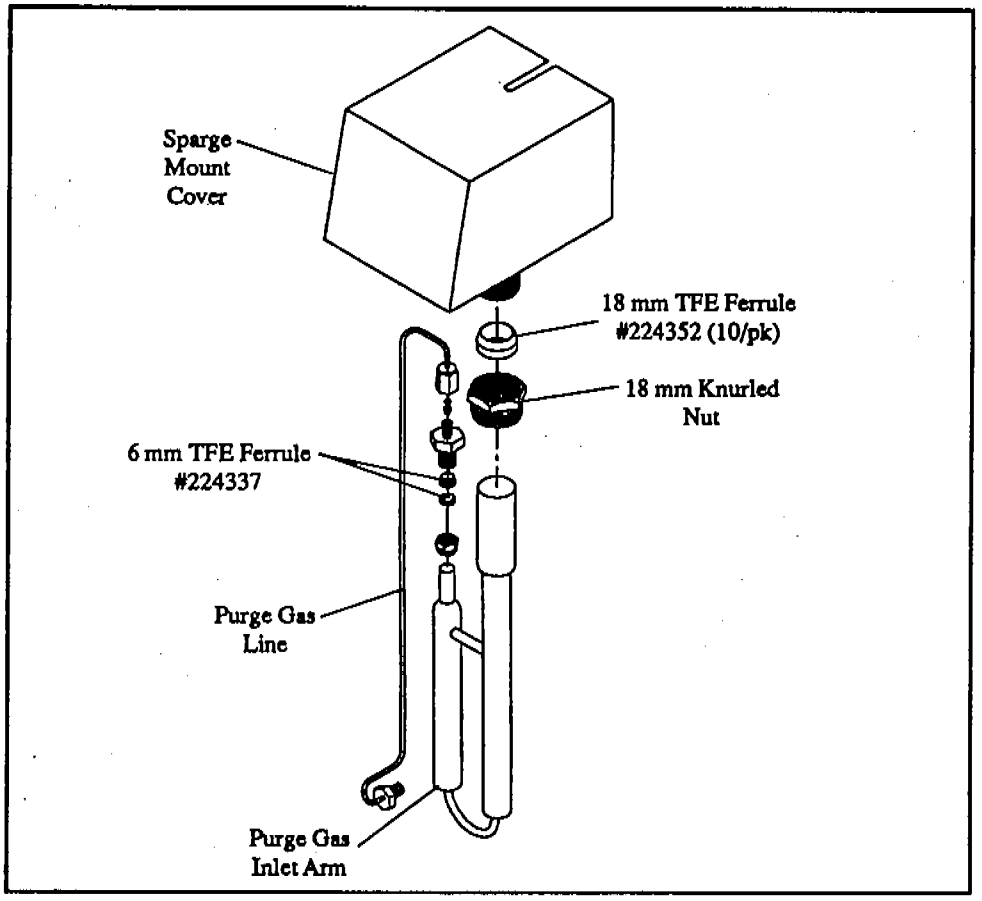


Fig. 3.1 Frit Sparger Assembly

Needle Sparging Setup

A Needle Sparge Hardware Kit is available as an option for the Sample Concentrator. A needle sparger may facilitate analyses performed on appropriate size samples of 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 methods using an optional sample heater.

- Remove the frit sparger from the 4560.

- Disconnect the frit-style Purge Gas Line from the front panel of the 4560.
- Replace with the needle-style Purge Gas Line provided and leak check fitting.
- Route the Purge Gas Line through the slot provided in the back left cover of the Sparge Door to the black 3-way Valve normally used for direct injection.
- Loosen the 3-way Valve from the bracket, rotate the Valve 90° clockwise, and re-tighten.
- Remove the Luer-lock Injection Adapter from its old position and thread the luer fitting into the frontmost port on the 3-way Valve.
- Connect the needle-style Purge Gas Line to the 9 o'clock position on the Valve and finger-tighten.
- Slide the appropriate needle sparger over the Purge Needle into the 18 mm Knurled Nut and finger-tighten. Leak check.

Gas Connections

- Connect the 1/8" Purge Gas Line between the purge gas source and the receptacle marked "Purge Gas Inlet" on the rear of the 4560. Use the standard 1/8" stainless steel or brass Swagelok™ nut and ferrule provided. If the Internal Carrier Flow Control Option is present on the 4560, this one connection is all that is necessary to provide purge and carrier gas to the analytical system. If the option is present, do not remove the plug from the port marked "Carrier Gas Inlet."
- If the Internal Carrier Flow Option is not present, a 1/16" stainless steel line must be connected between the GC Flow Controller (on the HP 5890, from the top of the "pencil" trap) and the "Carrier Inlet" on the rear of the 4560. The connection to the 4560 is made with a standard 1/16" Valco male nut and ferrule. The Carrier Gas Interface line for the HP 5890 GC is a standard part of the LDV Interface (OI Analytical Part #176900).

Electrical Connections

- Verify the voltage requirement (115 VAC or 230 VAC) for the unit you are installing. Provide power to the unit following the power requirements outlined in Chapter 1.
- Provide power to any OI Analytical Multisampler to be connected to the 4560.

Communication Cabling

The following figures are designed to depict communication cabling for several standard instrument configurations. Unless otherwise specified, all cables are OI Analytical cables. All figures show a rear view. **All units should be turned OFF when connecting the communication cabling.**

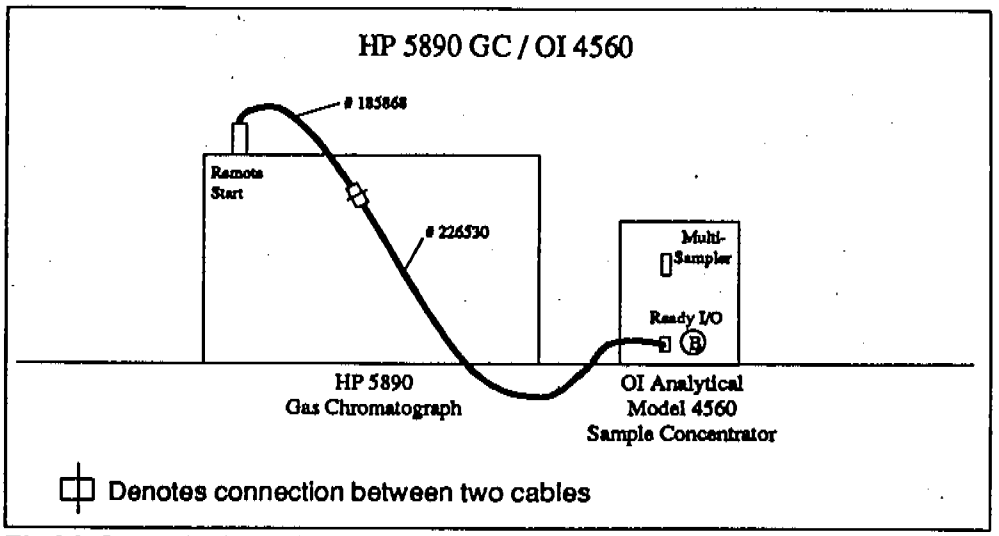


Fig. 3.2 Communication cabling between the HP 5890 and the OI 4560

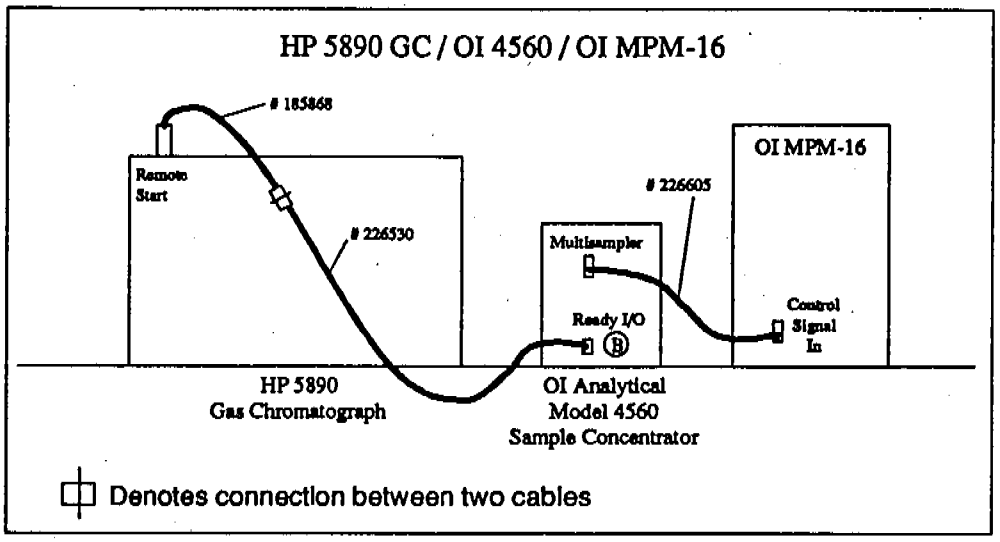


Fig. 3.3 Communication cabling between the HP 5890 /OI 4560 / OI MPM-16

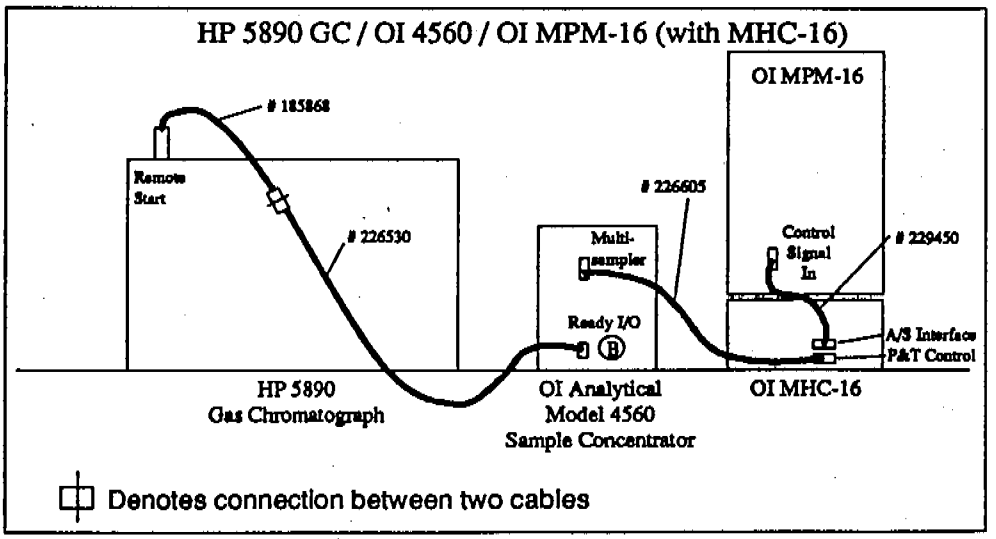


Fig. 3.4 Communication cabling between the HP 5890 / OI 4560 / OI MPM-16 (w/MHC-16)

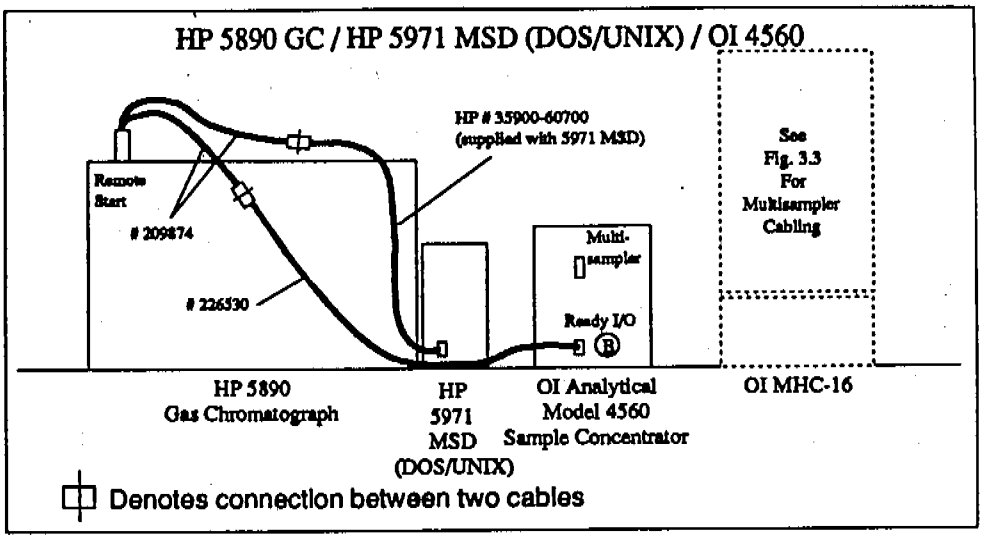


Fig. 3.5 Communication cabling between the HP 5890 / HP 5971 MSD (DOS/UNIX) / OI 4560

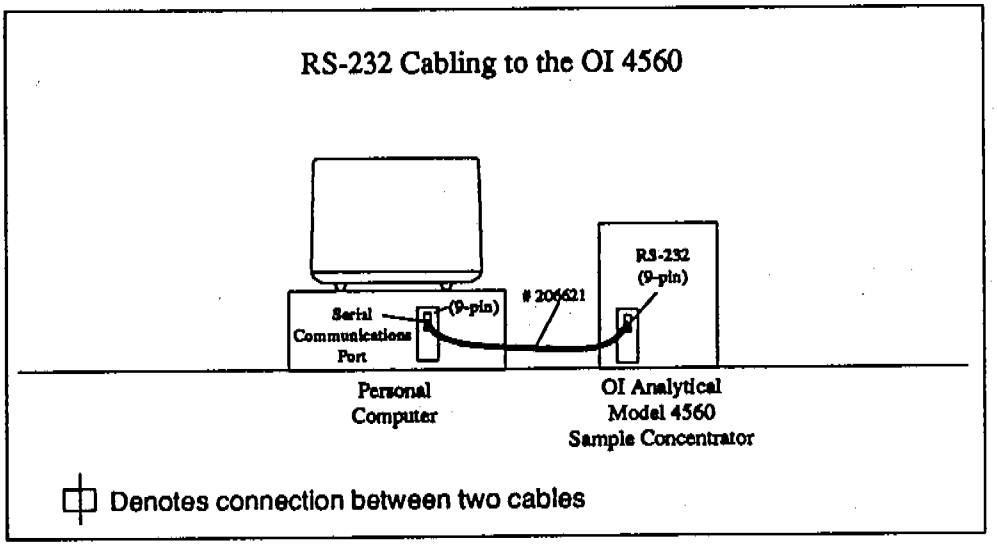


Fig. 3.6 Connection of serial communications port on PC to the OI 4560

All communication cables to the 4560 from external devices are located as follows or may be ordered separately when needed:

Cable #	Location
226530	Low-Dead-Volume Interface Kit HP 5890 (#176900)
185868	Low-Dead-Volume Interface Kit HP 5890 (#176900)
206621	PATTI®II Software Package
206605	MPM-16 Start-Up Kit
229450	MHC-16 Start-Up Kit
209874	Only sold separately when HP 5971 MSD is present

Transfer and Drain Line Connections

Transfer Line Connection to the GC Injector

The Heated Transfer Line on the 4560 can be connected directly to the analytical column via a union connecting the 1/16" Transfer Line to the appropriate column size. OI Analytical supplies a Low-Dead-Volume (LDV) Interface to make this connection and still retain direct injection capability onto the column. If you purchased an LDV Interface, refer to the *LDV Installation and Maintenance Guide* provided for proper installation; otherwise, interface the Transfer Line to the column minimizing dead volume.

Multisampler Transfer Line Connection to 4560

The Transfer Line from the MPM-16 Multisampler interfaces easily to the Sample Inlet Mount of the 4560. To connect the MPM-16 Transfer Line, follow these steps:

- Remove the Sparge Filter by turning it counter-clockwise with a 3/8" wrench.
- Replace the Filter with the A/S Interface Assembly (OI Analytical Part #209536) provided in the MPM-16 Start-Up Kit.
- Connect the MPM-16 Transfer Line to the A/S Interface adapter using the 1/16" Valco male nut and ferrule provided.

Drain Line Connection

The External Drain Line on the 4560 is made of urethane. To connect the Drain Line, press the Line supplied in the Start-Up Kit (OI Analytical Part #166224) onto the barbed Luer-lock fitting marked "drain" on the back panel of the 4560 and route to an appropriate drain or receptacle.

Upon completion of installation, advance to *4560 Functional Checks* in Chapter 4 to ensure proper function of the unit.

Chapter 4
Operation

This chapter provides basic information on the operation of the 4560 Sample Concentrator. The first section reviews individual states of operation to give the operator a fundamental understanding of purge-and-trap analysis using the 4560. The second section outlines functional checks that should be performed on the 4560 during start-up and then as required after installation. Finally, a detailed description of the 4560 keypad operation is provided.

The Model 4560 Sample Concentrator is designed to strip purgeable organic compounds from a water sample, concentrate them on a specified sequence of adsorbent materials, and rapidly desorb them using heat and carrier gas flow onto a gas chromatographic column. The 4560 should be regarded as one component of an analytical system for purge-and-trap analysis. Other necessary components include a gas chromatograph (GC), a 4560/GC Interface Kit, single or multiple detectors, and a data-handling device for quantification of detector signals. A multisampler for introducing samples to the concentrator may also be part of the overall analytical system.

The 4560 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 flow lines. By proper manipulation of valves and trap temperature through a specific time sequence, quantitative analysis of purgeable organics is achieved.

To help you understand how the 4560 has been designed, some basic operational concepts are outlined in this chapter.

Instrument States: The various sets of valve and trap temperature combinations defined for analysis using the 4560 are called states. The 11 primary states are called:

STANDBY	DRY PURGE
PURGE READY	DESORB READY
PRE-PURGE	DESORB (W/ or W/O DRAIN)
PRE-HEAT	BACKFLUSH BAKE (W/ or W/O PURGE)
PURGE (ADSORB)	DEPRESSURIZATION
PURGE COMPLETE	

The 4560 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 4560 has been programmed to allow the setting of up to 14 operator-defined sets of parameters called methods.

Each unit is shipped from the factory set to Method "1." Method 1 corresponds to parameters mandated by USEPA Method 502.2/524.2.

Sequencing through States

When the 4560 is operating in a run state, the microprocessor will advance the system through the following sequence of primary states, each existing for the time specified by the method, or for some states, until some other condition is met:

STANDBY - The 4560 will remain in STANDBY until all temperature setpoints have been attained. When all heated zones are in a ready state and the trap has cooled to the purge temp setpoint, the 4560 will advance to PURGE READY. STANDBY is one of several states not indicated by an LED on the front panel of the 4560 but is only seen on the display screen since no parameter entry is possible.

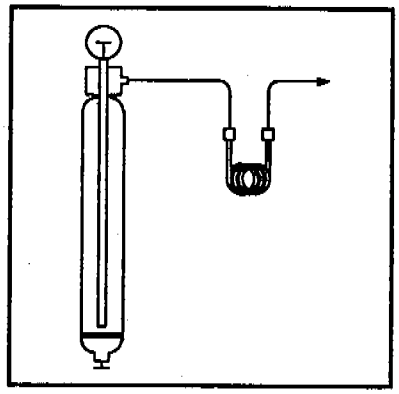


Fig. 4.1 Standby/Purge Ready

PURGE READY - This is a pass-through state that indicates to the operator that all conditions are met to start the next analysis. When manually syringe injecting into the 4560 (no multisampler is present), the unit can be configured to stop at PURGE READY after each sample run, waiting for the next manual injection and [START] to be pressed.

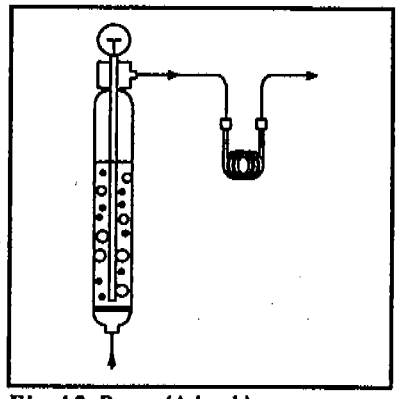


Fig. 4.2 Purge (Adsorb)

PRE-PURGE - This is an optional state normally used only for air analysis. Pre-purging an air-tube on the MPM-16 removes oxygen and excess moisture accumulated on the tube during sample collection. Pre-purging a hydrophobic substrate (e.g., Tenax®) reduces the amount of moisture transferred to the trap.

PRE-HEAT - This is a settable time prior to purge to allow a heated sample (i.e., air-tube, soil, or water) to equilibrate to a pre-set temperature before purge begins. This state is only used when an MHC-16 or 4560 Infra-Sparge Sample Heater option is present and sample heating is desired.

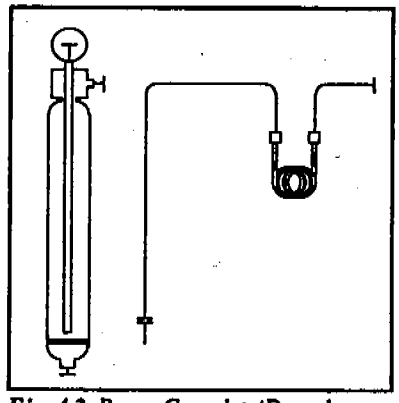


Fig. 4.3 Purge Complete/Desorb Ready

PURGE (ADSORB) - This is the sample extraction state. Inert gas is passed through the sample for a specified time and temperature, and volatiles are adsorbed onto the concentrator's trap for subsequent desorption to the GC column.

PURGE COMPLETE - This is a pass-through state indicating that the purge state is complete. If the water management system is "NOT READY," the 4560 will remain in PURGE COMPLETE until the Cyclone Water Management System (WMS) reaches the required setpoint.

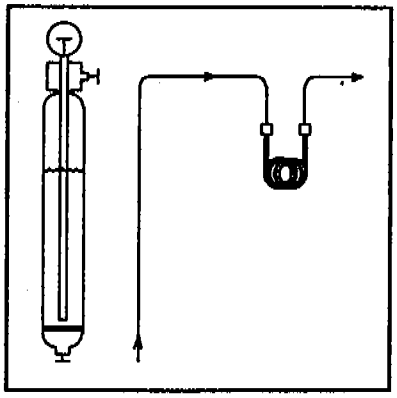


Fig. 4.4 Dry Purge

DRY PURGE - This is an optional state that is used to remove moisture from the hydrophobic substrates in the trap. In DRY-PURGE, the sparger is bypassed and the trap is purged with dry purge gas directly. DRY-PURGE is

usually not necessary when an effective water management system is present.

DESORB READY - After all purging (PURGE and DRY-PURGE) is complete, the 4560 will advance to DESORB READY. DESORB READY is a diagnostic state in which the 4560 checks for ready signals from other components in the system (i.e., GC) before advancing to DESORB.

DESORB - During DESORB, the Concentrator's trap heats rapidly to the set temperature, transferring volatile components through the heated transfer line of the 4560 to the GC injector port. If the MPM-16 is used (draining is not possible) and the 4560 is configured not to drain, no sample drain will be attempted (DESORB WITHOUT DRAIN). Otherwise, an aqueous sample is automatically drained during the DESORB state (DESORB WITH DRAIN).

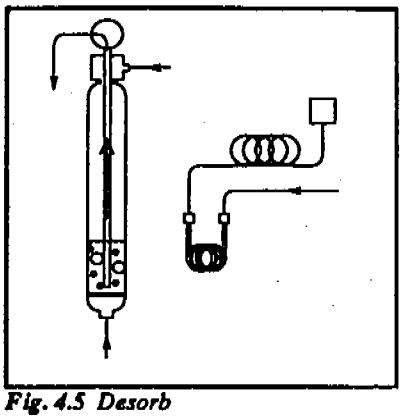


Fig. 4.5 Desorb

BACKFLUSH BAKE - This clean-up state backflushes the trap under heat and reverse flow to remove and vent any components not transferred to the GC column. One may either purge or not purge the sample during BAKE (see *Configuration Key*).

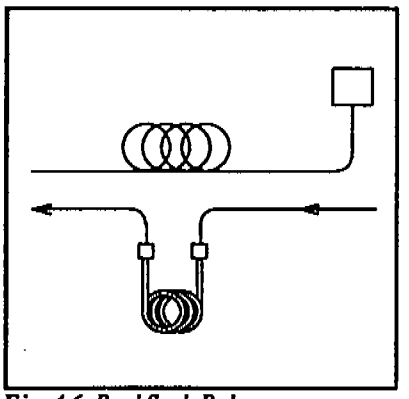


Fig. 4.6 Backflush Bake

DEPRESSURIZATION - This state is active only when the MPM-16 is ON in the Configuration menu. Just after BAKE, before the multisampler advances, pressure in the vessel above the sample is vented to prevent backstreaming of sample into the purge needle.

General State Guidelines

Illuminated *orange* lights in the Cycle State Indicator show which states have been activated during a run (see Fig. 4.7).

Illuminated *green* lights in the Cycle State Indicator show the current state (see Fig. 4.7).

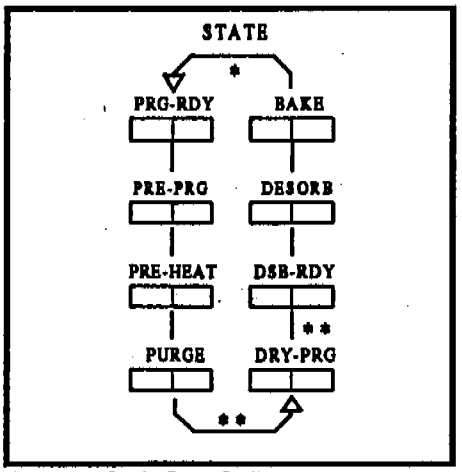


Fig. 4.7 Cycle State Indicator

* Two states occur between BAKE and PURGE READY that are not shown on the cycle state indicator:

1. DEPRESSR, which occurs after BAKE when the MPM-16 is present in the Configuration Menu to depressurize the sparger for 0.50 min. (See *Sequencing Through States* in this Chapter.)

2. STANDBY, in which trap temperature is equilibrating to the PURGE set temperature.

** A PURGE COMPLETE state occurs between PURGE and DESORB READY or DRY PURGE and DESORB READY if a DRY PURGE time is entered. The water management system (WMS) may be cooling before DESORB during this state.

4560 Functional Checks

This section includes step-by-step procedures for confirming the proper function of the 4560 before sample analysis. Refer to Chapter 5, *Maintenance and Trouble-shooting*, for explanations of how each of these checks can be used to isolate problems.

Pre-Power-Up Checks

- Adjust purge and carrier gas pressures (purge pressure should be set to 20 psi in STANDBY or PURGE READY).
- Confirm that carrier gas flow is set to desired rate.
- Turn on power switch. The Model 4560 will perform a self-diagnostic test for 1 min.

Performing a Self-Test

The 4560 will perform a self-diagnostic test on power-up. The self-test changes the setpoint for the heated zones to 25° C above ambient. At the end of the self-test, the readings for all of the zones will be compared to the setpoint (ambient +25° C) and the error for each zone will be computed internally. Heated zones tested are listed below:

Trap
External Heater
Sample Inlet
Control Manifold
Sample Heater
Valve
Transfer Line

Any zone that is not within its valid range at the end of the self-test will cause a reported error and self-test failure.

NOTE: A zone will not be included in the self-test if it was turned OFF the last time the unit was in operation.

The zones included in the test are dependent upon whether the unit is powered up "cold" or "warm." All zones will be included on a cold start power-up condition. If a zone reading is more than 25° C above ambient on power-up, it will not be included in the self-test. This is considered a warm start.

The following conditions can cause a zone to fail the self-test.

1. A safety switch will not allow a zone to heat (i.e., trap and/or sample heater).

2. An open thermocouple (TC) connection has caused an over-temperature condition. If the over temp LED on the AC/IO board is "ON," power will be shut off to all heated zones. Unused TC connectors should be shorted and the corresponding zone shut off through the keyboard.

3. The fuse for the heated zone is blown.

4. Thermocouple is shorted for the zone that has failed. The zone will heat, but readings never change.

If the unit fails the self-test, you will be prompted to press [2nd] [9] for a display of the errors or failure conditions. Pressing [2nd] [9] displays the number of errors found, followed by a prompt to press [ON] or [OFF]. Pressing [ON] or [OFF] displays the error if only one error occurred, or scrolls through multiple error messages.

If a hardware failure for the electronics is detected, check the cabling connections between the IO Board and CPU Card to make sure they are securely in place. Call OI Analytical Technical Support at 1-800-336-1911 if the cabling seems correct and the unit still does not pass the self-test.

The self-test can be bypassed at any time by pressing any key on the 4560 during the test. At the end of the self-test, the unit will restore all settings to their previous values.

Confirming 6-port Trap Valve Rotation

- Press [2nd] [1] to show 6-port Valve position.

- Test 6-port Valve rotation by toggling Trap to GC [ON] and [OFF]. Trap to GC ON = Desorb. Trap to GC OFF = Purge.

- Verify that you hear the Valve actuate.

Setting Purge Flow Rate

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

- While the system is in STANDBY or PURGE READY, set the purge gas pressure to read 20 psi, as indicated on the purge pressure gauge on the front panel of the 4560.

- Confirm that the 4560 is not in a "RUN" state (i.e., the green "RUN" LED is OFF). If the "RUN" LED is ON, press [HOLD] to exit the "RUN" state.
- Press [OFF] to advance to PURGE.
- Measure the purge flow out of the Purge Vent under the sparger on the front of the 4560, using a flow meter accurate to within 1 ml/min.
- Adjust to desired flow rate using the Purge Flow Controller on the front panel of the 4560.
- Confirm that the purge pressure in PURGE is 5-8 psi.

NOTE: Purge flow rate can have significant effect on the recovery and extraction efficiency of some analytes. Experimentation with purge flow rate is an important factor in optimizing your analysis.

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.
- Stop purge flow from Bake Valve by plugging the end of the Vent Line using a septum.
- 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 an electronic gas-leak detector, if necessary, to find leaks.
- With the system pressurized at 20 psi (i.e., the Vent plugged), turn the Purge Pressure Regulator a few turns counter-clockwise. If the system is leak-free, the purge pressure on the gauge should not drop >1 psi over a 1 min period.
- Release flow restriction to regain purge flow.
- Return to STANDBY by pressing [2nd] [ON].
- Re-adjust purge pressure to 20 psi.
- Press [OFF] to advance to PURGE again.
- Confirm return of purge gas pressure to 5-8 psi.

Drain Check

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

- Confirm purge flow rate and leak-tightness per above, leaving sample in purge vessel.
- Confirm proper Sample Valve position for Drain (see Fig. 4.8, p. 32). Press [2nd] [CLEAR] to drain. You will be asked to verify drain by pressing [ENTER].

- Measure how long it takes to drain the 5 ml sample. For a 5 ml sample, about 8-10 sec is normal. If drain time is significantly slower than this, inadequate gas flow to the purge vessel, improper Sample Valve Handle position, or a restriction in the Drain Line is probable. 30 sec is allocated for draining (0.50 min).

Carrier Gas Leak Check

- Disconnect Transfer Line from GC Injection Port.
- Cover end of Transfer Line with a septum to completely stop flow.
- Observe that the GC Flow Controller pressure (through gauge on GC or front of 4560) 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

- Press [2nd] [CLEAR] [ENTER] as described in *Drain Check*.
- Confirm that the Trap temperature rises rapidly to the Bake Temp setpoint by watching the temperature display. Any sample that is in the purge vessel will automatically drain, then the unit will cycle to BAKE.
- The system will bake the Trap until BAKE time runs out, or [HOLD] may be pressed during a "RUN" for extended baking.
- After Trap BAKE is complete, advance the system to STANDBY by pressing [2nd] [ON]. The system will return to STANDBY automatically.

Running A Sample

The preceding functional checks should be completed before attempting to analyze a sample. It is also assumed that the GC Column and detector have been properly interfaced, conditioned, and are ready for sample introduction. 4560 operating procedures are as follows:

Syringe Sample Loading

- Allow sample to come to ambient temperature before introducing it to the syringe.
- Remove the plunger from the sample syringe.
- Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Place your finger over the end of the syringe to hold the sample in the barrel.
- 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 using a 10 µl syringe, then close the valve.

Syringe Sample Injection

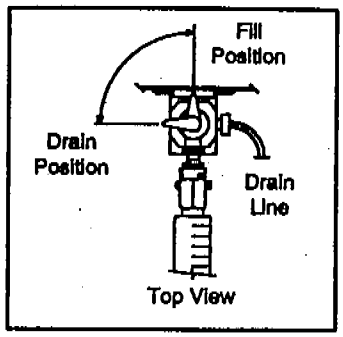


Fig 4.8 Sample Valve

- When the system is in PURGE READY, press the [START] key to start the analysis.
- Attach the syringe with valve to the 4560 Sample Valve Syringe Port.
- Open the syringe valve, then turn the Sample Valve Handle to the fill position (see Figure 4.8).
- 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.
- 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.
- 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.

Needle Sparging

The instructions given here are to be used only as guidelines. Actual methodology may require different parameters (weights, volume, etc.) than stated.

Purge-and-trap analysis using a needle sparger is similar to operation with a frit sparger. There are 2 main differences, however. First is the purging efficiency of a needle sparging apparatus which, in general, is lower than that for a frit (or dispersed bubble) sparger. 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.

The following two sections describe the analysis of liquid samples and solid samples, respectively, when using a needle sparger on the 4560.

Instructions for Needle Sparging Liquid Samples

- Disable the drain on the 4560.
- Follow the instructions outlined earlier for the proper syringe sample loading technique.
- Attach the syringe to the 4560 Sample Valve Syringe Port.
- Turn the Sample Valve Handle to the "Fill" position.

- Inject the sample into the purge vessel and close the Sample Valve.
- Press the [START] key to begin the analysis. The sample will begin purging for the time determined by the purge time setpoint and continue through the other states according to the setpoint values.
- After the 4560 cycles to BAKE, loosen the lower Sparger Nut, and slide the purge vessel off.
- Clean and rinse the vessel before re-installing or install a cleaned and baked purge vessel and tighten the lower Nut finger-tight.
- When the 4560 cycles to PURGE READY, proceed with the next sample as above.

Instructions for Needle Sparging Solid or Sludge Samples

- Weigh a clean dry purge 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 g sample is typical, depending on expected analyte concentration.
- Install the purge vessel with sample on the 4560 with the neck of the purge vessel inserted properly into the Sparger Nut. Tighten the Sparger Nut finger-tight for a leak-free seal.
- If a heated sparge is to be performed, set the Infra-Sparge Sample Heater to the desired setpoint.
- 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*).
- Attach the syringe to the 4560 Sample Syringe Port.
- Turn the Sample Valve Handle to the "Fill" position.
- Inject the reagent water into the purge vessel and close the Sample Valve.
- Press the [START] 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 4560 cycles to BAKE, loosen the Sparger Nut and slide the purge vessel off.
- Clean, rinse, and dry the vessel before weighing the next sample, or perform the subsequent analysis with a cleaned and baked vessel.
- When the 4560 cycles to PURGE READY, proceed with the next sample as above.

Key Description

The keypad of the Model 4560 Sample Concentrator contains eight key types: Temperature Keys, Time Keys, Number Keys, Action Keys, Function Keys, Multisampling Keys, a Configuration Key, and a File Management Key. Some parameters are accessed by simply pressing the appropriately labeled key, while others are accessed using 2nd Function Keys. The 2nd Function Key parameters are labeled in gold writing above the keys. For example, Pre-Purge is a 2nd Function Key. **2nd Function keys are enabled by pressing the gold key [2nd] and then pressing the desired 2nd Function selection.**

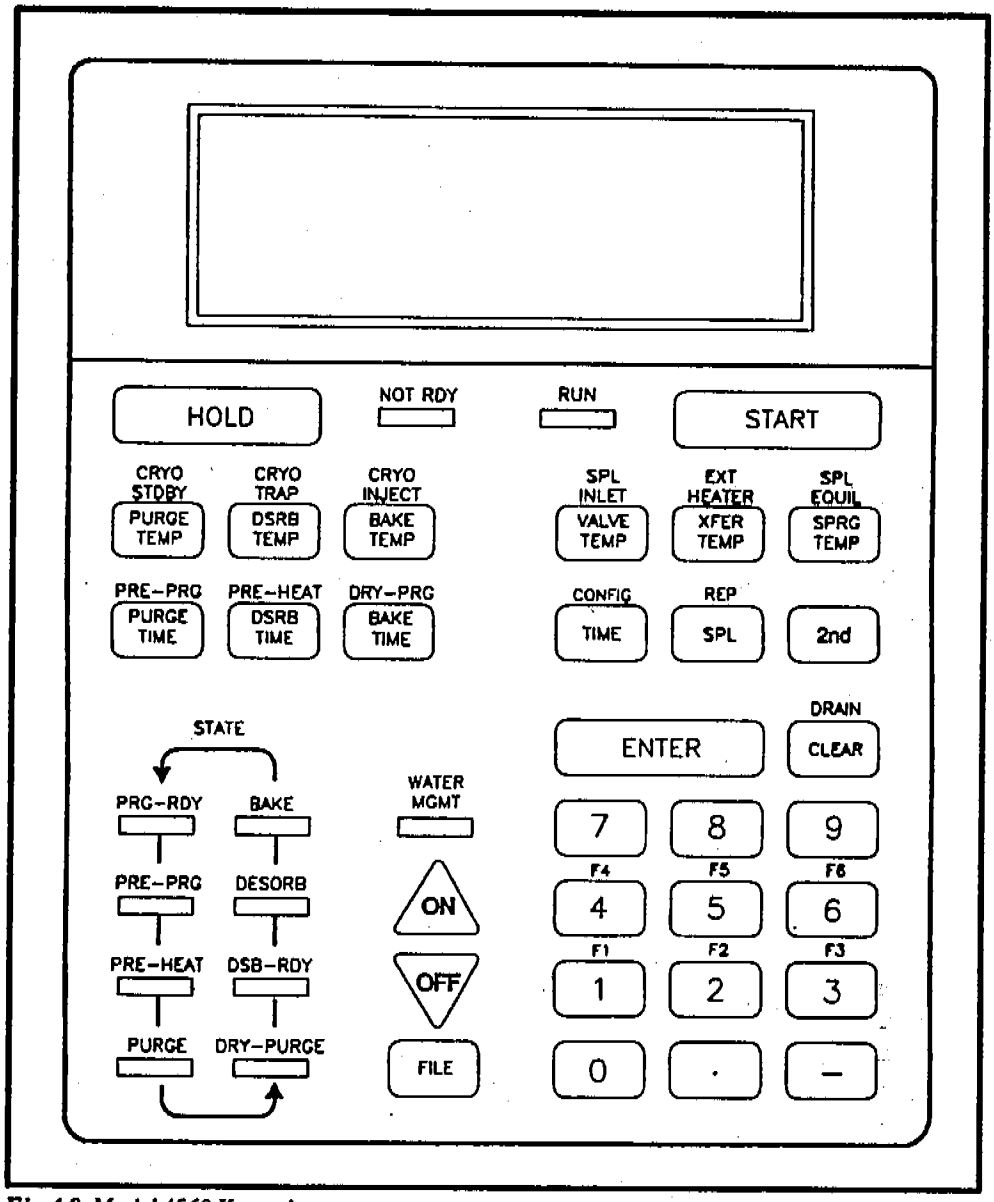


Fig 4.9 Model 4560 Keypad

Temperature Keys

Eight temperature zones are definable on the 4560. To set any of the following types of temperatures, press the key identified below in bold print to display the setting, enter the desired temperature (within the prescribed range), and press [ENTER].

Purge Temp (set from ambient to 300° C) indicates the temperature of the Trap during PURGE. It is usually set at 20° C (default setting).

Desorb (DSRB) Temp (DSRB TEMP) (set from ambient to 300° C) indicates the temperature of the Trap during DESORB. It is usually set to 180° C (default setting).

Bake Temp (set from ambient to 300° C) indicates the temperature of the Trap during BAKE. It is usually set to 200° C (default setting).

Valve Temp (set from ambient to 300° C) indicates the temperature of the Valve Oven at all times during the cycle. It is usually set to 100° C (default setting).

Xfer Temp (set from ambient to 200° C) indicates the temperature of the Transfer Line at all times during the cycle. It is usually set to 100° C (default setting).

Sparge Temp (set from ambient to 100° C) indicates the actual temperature of the sample (if the Sample Heater is present). The set sparge temperature (sample temperature) is maintained in all states except STANDBY, PURGE READY, and PRE-PURGE. This key controls the optional Sample Heater. *It should be OFF if the optional Sample Heater is not present. The sparge temperature defaults to 20° if this zone is ON.*

Spl Inlet [2nd Function of **Valve Temp**] (set from ambient to 200° C) indicates the temperature of the Sparge Mount. It is usually set to 40° C. This is only functional if the Sample Heater Option is present.

Ext Heater [2nd Function of **Xfer Temp**] (set from ambient to 200° C) indicates the temperature of the MPM-16 Valve and Transfer Lines or another external device controlled through the External Heater circuit. If no external device is present, this should be OFF. If ON, the default temperature is 100° C.

If a heated zone is over its prescribed set temperature (or has an open thermocouple), a series of 4 warning beeps will be heard and an "Overtemperature Detected" message will be displayed. The situation should be corrected before further operation is attempted.

NOTE: The 4560 CPU will terminate power to all heated zones when an "overtemp" is detected.

Time Keys

Seven Time functions are definable on the 4560. To set any of the following times, press the key identified in bold print to display the setting, enter the desired time (0 to 999.99 min), and press [ENTER].

Purge Time indicates the duration of PURGE. It is usually set to 11 min (default setting).

Desorb (DSRB) Time indicates the duration of the DESORB state. It is usually set to 4 min (default setting).

Bake Time indicates the duration of the BAKE state. It is usually set to 20 min (default setting).

Pre-Purge (PRE-PRG) [2nd Function of **Purge Time**] indicates duration of PRE-PURGE. It is usually set to 0 (default setting).

Pre-Heat [2nd Function of **Desorb Time**] indicates duration of PRE-HEAT. It is usually set to 0 (default setting).

Dry-Purge (DRY-PRG) [2nd Function of **Bake Time**] indicates duration of DRY-PURGE. It is usually set to 0 (default setting). Dry-Purge is not often used due to the presence of Cyclone Water Management™.

Time a. when pressed *once* indicates the duration of current run (elapsed time).

b. when pressed *twice* indicates the remaining time of current run.

c. when pressed three consecutive times, a timer is displayed to aid in flow setting. To start and stop the timer, press [ENTER]. Reset the timer by pressing [CLEAR].

Number Keys

Temperatures and times can be set on the 4560 by using the Number Keys (0-9).

Action Keys

The [ENTER] key terminates numeric entry. After [ENTER] is pressed, the value is verified to ensure that it is acceptable. If the value is not acceptable, another value may be entered immediately.

An asterisk (*) flashes as a new value is entered. This indicates that an entry is in progress.

Press the [START] key to begin a run or to continue a run that was interrupted (see [HOLD]. If a sequence is entered and sequencing is on, [START] is also used to initiate the sequence.

To pause a run, press the **[HOLD]** key. The [HOLD] key will stop elapsed time. Restart elapsed time by pressing [START].

NOTE: Only the time is stopped, the unit will continue executing the current state (i.e., PURGE).

Abort a run and return to STANDBY by pressing the gold key [2nd] and then [ON]. Elapsed time must first be paused by pressing [HOLD].

Advance to PURGE COMPLETE automatically from any state by pressing the gold key [2nd] and then [OFF].

Manual **Drain** (2nd Function of [CLEAR]) drains any sample in the purge vessel (if the sample valve is set to drain) for .50 min, then advances the unit to BAKE for the duration of the BAKE time. If the unit is running, elapsed time must be stopped [HOLD] before draining is allowed. [ENTER] must be pressed after [2nd] [CLEAR] to verify the drain command.

Press the [ON] or [OFF] keys to manually advance or reverse through the enabled states with no elapsed time running. [ON] and [OFF] keys are also used to do the following:

a. Enable or disable a heated zone.

Example: if [VALVE TEMP] is pressed, then the following will be displayed

if [OFF] is then pressed, then the following will be displayed

No power will be provided to the selected zone until the zone is turned back ON.

b. Select between options.

Example: if the gold key [2nd] and then [TIME] are pressed, then the following is displayed

The flashing line under "States" identifies it as the selected item if [ENTER] is pressed.

if [OFF] is then pressed, the following will be displayed

"Options" is now the selected option if [ENTER] is pressed.

c. Enable or disable an option or state.

Continuing the above example:

if [ENTER] is pressed, then the following will be displayed

if [ON] is pressed, then the following will be displayed

The [CLEAR] key, if pressed when no setpoint entry is in progress, displays Model 4560 readiness. The [CLEAR] key is also used during setpoint entry (before pressing [ENTER]) to erase an entry in progress. When [CLEAR] is pressed (if elapsed time is not running), parameters "Not Ready" and parameters that have been turned OFF scroll indefinitely on the display. If all parameters are ON and "Ready," a "System Ready" message is displayed. If a run is in progress, messages scroll once and then the current state is displayed. As the purge-and-trap cycle is executed, the display shows the current state, time, and trap temperature. If another key is pressed, the selected parameter is displayed for 5 sec and the display then reverts back to the current state.

Function Keys

[F1] 6-port Valve Rotation [2nd] [1]

The [F1] key allows the operator to manually rotate the 6-port Valve when no elapsed time is running.

Trap to GC ON = DESORB
Trap to GC OFF = PURGE

F1 is used to manually place the 4560 Trap in-line with the GC (Trap to GC ON) or out-of-line with the GC (Trap to GC OFF). The Valve is manually rotated (the rotation can be heard) by pressing the [ON] or [OFF] key. If the Trap temperature is above 50° C when manual Valve rotation is attempted, the following message will appear.

This is a safety precaution, preventing the absence of flow through a heated Trap and subsequent damage to the trapping substrate.

[F2] Auxiliary Thermocouple [2nd] [2]

The Auxiliary Thermocouple can be used to signal the 4560 to advance to DESORB at a settable temperature when the normal contact closures signaling "GC Ready" to the 4560 are not available.

Pressing [2nd] [2] displays the following

The first line displays the temperature reading for the Auxiliary Thermocouple input. If the auxiliary zone is turned ON, the 4560 will advance to DESORB from DESORB READY when the GC oven temperature is at or below the temperature entered on the

second line. Auxiliary should be normally OFF when the GC Ready contact closure is available from the GC. A Type-K Auxiliary Oven Thermocouple is available from OI Analytical, if needed.

[F3] See *Method Sequencing with the 4560.*

[F4] Control Manifold [2nd] [4]

The Control (Valve) Manifold is the aluminum block in the left bay of the 4560 that heats the Purge, Dry-Purge, and Bake Valves to a preset 50° C and directs flow between these Valves. This can cause a self-test failure if it is not heating properly and can therefore be monitored by pressing [2nd] [4]. If a problem occurs with this heated zone, call the OI Analytical Technical Support Toll-Free Hotline at 1-800-336-1911.

[F5] Sparger Size [2nd] [5]

When the optional sample Heater on the 4560 is present, the heating ramp differs depending on sample size (5 or 25 ml). Pressing [2nd] [5] displays the following

Selecting the appropriate sparger size ensures ±1° C temperature control for each sample size. Select between sample sizes by pressing [ON] or [OFF]; pressing [ENTER] is not necessary to verify entry.

NOTE: Selecting 25 ml when a 5 ml sample is being heated can lead to temperature overshoot.

This entry has no effect when the Sample Heater Option is not present.

Multisampling Keys

SPL displays the number of samples (0-999) to be run (Final Spl) and the present sample being run (This Spl). For continuous cycling, set Final Spl to 0.

Rep [2nd Function of SPL] indicates the number of replicates per sample. This key can be used to run up to 99 replicates of a single sample. Normally, with discrete samplers, # replicates = 1. Current replicate is used as a counter.

If the unit is configured for multisampling (i.e., PURGE READY INPUT = OFF), the 4560 will continue to cycle until This Spl = Final Spl.

NOTE: When the 4560 is configured for the MPM-16, the actual sample position may be read to the far right of the Spl screen. This number in no way influences the setting of Final Spl or This Spl but only monitors Valve position on the MPM-16.

Method Sequencing with the 4560

The 4560 can internally download up to 5 selected methods during any automated (multisampler) run. For example, if you have 15 samples to run on the MPM-16, but not all by the same Sample Concentrator method, you may select up to 5 different saved methods and specify the number of samples to be run under each method.

[F3] Sequencing [2nd] [3]

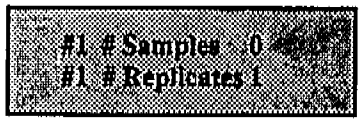
The unit will display the Main Sequencing Screen

The OFF designation indicates that sequencing is currently inactive and that the unit will run the currently loaded method until a new method is *manually* downloaded. Numbers [1] - [5] each represent method entries that compose the sequence.

When [1] is pressed, the unit will display

Select the first method to be run in the sequence by scrolling through the list using the [ON] or [OFF] key and pressing [ENTER].

The unit will display



(Indicates Method #1 selected)

Enter the number of samples to be run under the method and press [ENTER]. Press [OFF] to move to number of replicates.

NOTE: When using a discrete multisampler like the MPM-16, replicates are only applicable for carryover studies, purge efficiency tests, etc. Normally, using the MPM-16, the number of replicates should be set to 1.

Enter the number of replicates and press [ENTER]. Verify the number of samples and replicates you have entered and press [CLEAR].

Back at the Main Sequencing Screen, repeat above steps for methods [2] - [5], as necessary.

Once complete, from the Main Sequencing Screen, press [ON] to activate sequencing. The number of methods entered will be verified on the display:

Press [CLEAR] to exit sequencing.

NOTE: When sequencing is activated, parameters for method [1] are automatically downloaded; however, the sequence does not begin until [START] is pressed.

If a sample in the sequence is aborted for any reason, the sequence may be continued at the same sample position by pressing [START] again. A sequence may be reset by returning to the sequencing display [2nd] [3], pressing [OFF] then [ON], exiting sequencing (press [CLEAR]), and pressing [START] once again.

Other notes on sequencing:

- A sequence always will start from the current multisampler position.
- The current method number of samples/number of replicates can be accessed through the [SPL/REP] key on the front panel.

Configuration Key

[Config] (2nd Function of [TIME]) allows the analyst to identify and configure options for the 4560 (e.g., MPM-16, CFM) *or* define **States** to override the **Option** configuration. Use [ON] / [OFF] Keys to select between **States** or **Options** and press [ENTER].

States allows the analyst to override option configuration. Use [ON] or [OFF] keys to scroll between **Inputs, Outputs, and Flows.**

> **Inputs** allows the analyst to enable or disable PURGE READY and/or DESORB READY input requirements.
>
> **Outputs** allows the analyst to define outputs given by the 4560 at the start of DESORB and start of BAKE.
>
> **Flows** allows the analyst to choose whether to drain the sample during DESORB or purge the sample during BAKE.

Options allows the operator to auto-configure the 4560 to accommodate external OI Analytical devices such as the MPM-16 and the CFM. Pressing [ENTER] scrolls through each configuration option; [ON] / [OFF] enables or disables the option.

> For example, to configure the 4560 for the MPM-16 and CFM, perform the following steps:
>
> Press [2nd] [TIME]. The following will be displayed

Press [OFF]. The following will be displayed

Press [ENTER]. The following will be displayed

Press [ON]. The following will be displayed

Press [ENTER]. The following will be displayed

Press [ON]. The following will be displayed

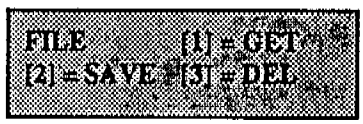
Press [CLEAR] to exit the Config Menu.

The Config selections are included in saved methods, but not in pre-stored Method 502.2/524.2, as described in the next section.

File Management Key

[File] Key is to GET, SAVE, and Delete (DEL) method files used in the 4560. Upon pressing [FILE], the File Menu is displayed. GET, SAVE, and DEL can be accessed by selecting the appropriate input number key.

File Menu Display



Save File allows setpoints to be saved under a specific name using the [FILE] key followed by the [2] key. The SAVE FILE display appears and a file can then be selected using the [ON] and [OFF] keys. Saving new setpoints over an existing file replaces all setpoints currently defined in the selected file.

Once the file is selected, by scrolling with the [ON] / [OFF] Keys, the file is named and then saved by one of the following 3 methods:

- Use the numeric keypad to name an undefined file or rename an existing file, then press the [ENTER] key to save the setpoints. When a file is named, but not

saved within 5 sec by pressing [ENTER], the 4560 defaults to the File Menu Display.

- Use the [ENTER] key only to name and save a file as its file number if the file is undefined. For example, selecting the undefined file number 7 then pressing [ENTER] would save the file as "FILE 7."

- Use the [ENTER] key only to retain a file name from a previously named file. For example, selecting the existing file "2: 601/602" then pressing [ENTER] retains the file name and replaces all current setpoints under file "2: 601/602."

When using the [ENTER] key to save a file, one of two messages will be displayed. The message "FILE SAVED" indicates the new setpoints were saved under the selected file. The message "FILE NOT CHANGED" indicates no setpoints were changed to the selected file.

Get File allows saved files to be retrieved by pressing the [FILE] key followed by the [1] key. Select a file using the [ON] and [OFF] keys followed by [ENTER] to load the file or [CLEAR] to return to the file menu. One of the following three messages will be displayed upon pressing [ENTER]:

- FILE LOADED - The selected file setpoints have been successfully loaded and are now current.

- NO FILE HERE - A file was selected that contained no setpoints (i.e., an undefined file).

- SAVE CURRENT SETTINGS? Y/N - Indicates setpoints have been changed but not saved. Use the [ON] and [OFF] keys to select either Y (yes) or N (no) followed by [ENTER]. Selecting N ignores the changed setpoints and loads the selected file. Selecting Y allows the changed setpoints to be saved under a new or existing file. The screen display changes to the SAVE FILE mode. Once the setpoints are saved, the selected file is loaded.

Del File allows setpoints to be deleted using the [FILE] key followed by the [3] key. Select a file using the [ON] and [OFF] keys followed by [ENTER]. The file will then become undefined (i.e., the file name and all setpoints are erased). If File 11 is deleted, only the setpoints are erased, the file name is retained.

The setpoints for EPA Method 502.2/524.2 are stored as a protected file in file position 1. This file cannot be deleted or overwritten. The 502.2/524.2 file, *unlike* all other saved files, does not contain configuration parameters. This allows the operator to default back to 502.2 parameters without changing the system configuration.

Other Keystrokes

Keyboard Lock [CLEAR] [.] [-]

To ensure that no temperatures or times are changed on the 4560, a keyboard lock is available by pressing [CLEAR] [.] [-] and then [ENTER]. Selecting [ON] prohibits parameter entry (only allows monitoring), until the keyboard lock is removed. Remove the keyboard lock through the same keystroke entry and selecting [OFF].

Group ID [CLEAR] [.]

Several 4560s can be linked together for RS-232 communication using the datalink port on the CPU Card. Several 4560s can then be controlled with PATTI®II Windows™ Software for full Sample Concentrator control.

[CLEAR] [.] shows the group ID used to identify the 4560 in hexadecimal and may be changed to another value by pressing [ON] or [OFF] to scroll through a list of IDs. Press [ENTER] to confirm an ID.

Chapter 5
Maintenance and Troubleshooting

Maintenance

The only components of the 4560 that require periodic maintenance are the Trap, purge vessel (sparger), and Sparge Filter. 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 a need for increased pressure 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, manually place the system in BAKE and set the Bake Temp to 200° C. 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.

Changing the Trap

- If possible, turn the 4560 off and unplug the unit when changing the Trap.
- Open Trap Access Door located in the back left cover of the unit. A safety switch will automatically cut power to the Trap when the door is opened if the unit has not been turned off.
- Unplug Thermocouple.
- Press in on both sides of the stainless steel housing that covers the Trap and remove the housing by lifting upward.
- Loosen both fittings of the Trap with a 3/8" wrench and remove the existing Trap. It is not necessary to use a back-up wrench on the Bulkhead when loosening the fittings.
- Install the new Trap with the outlet (designated by an "O" on the Trap Nut) connected to the Bulkhead closest to the center wall of the unit. The inlet will provide the trap type (#7 - #9) on the Nut.
- Tighten the Trap Nut finger-tight plus 1/2 to 3/4 of a turn.
- Plug in Thermocouple.
- Replace the stainless steel Trap cover and close the Trap Access Door.
- Perform a leak check.

Cleaning/Changing the Sparger

The 4560 is designed to accommodate either frit or needle-style spargers (5 or 25 ml) with little change of hardware. To clean a sparger, rinse thoroughly several times with ultrapure water and bake in an oven following normal laboratory procedures. Only use

solvents when water does not clean sufficiently. Use a solvent that will not be detected by your detector, if possible. To install the sparger, follow these steps:

- Decide whether you will be using the frit or needle-style sparger.

To Change the Frit Sparger

- Loosen the top nut of the Brass Fitting on the Purge Gas Inlet Arm of the sparger.
- Remove the stainless steel Purge Gas Line from the fitting located on the front panel, just under the sparger.
- ***CAUTION: The 18 mm Knurled Nut may be hot!*** Loosen the 18 mm Knurled Nut securing the frit sparger to the bottom of the Sparge Mount Block. This fitting should be able to be loosened by hand.
- Lower the sparger until it clears the Purge Needle.

To Install New Sparger

- Place new sparger over the Needle and insert neck into 18 mm Fitting.
- Insert 1/16" stainless steel Purge Gas Line into Brass Fitting on Purge Inlet Arm of the sparger.
- Tighten both fittings securing the sparger and leak check.

To Convert to a Needle-Style Vessel

- Remove the frit sparger as described above.
- Disconnect the frit-style Purge Gas Line from the front panel of the 4560.
- Replace with the needle-style Purge Gas Line (OI Analytical Part #227389) and leak check fitting.
- Route the Line through the slot provided in the back left cover of the Purge Vessel Door to the black 3-way Sample Valve normally used for direct injection.
- Loosen the 3-way Sample Valve from the bracket, rotate the Valve 90° clockwise, and re-tighten.
- Remove the Luer-lock Injection Adapter from its old position and thread the Luer fitting into the frontmost port on the 3-way Sample Valve.
- Connect the needle-style Purge Gas Line to the 9 o'clock position on the Valve and finger-tighten.
- Slide the appropriate needle sparger over the Purge Needle into the 18 mm Knurled Nut and finger-tighten. Leak check.

Replacing/Maintaining the Sparge Filter

- The Sparge Filter, located on the left side of the Sparge Mount Block, may be cleaned or replaced, as necessary.
- Remove the Filter by turning counter-clockwise with a 3/8" wrench.
- The Filter may be cleaned by rinsing it with methanol and drying with ultrapure nitrogen or helium.
- The Filter may optionally be baked at 100-150° C for 30 min.
- Replace the Filter by tightening finger-tight plus 1/4 to 1/2 of a turn.

4560 Diagnostic Tools

Troubleshooting the 4560 has been condensed to a set of generic step-by-step procedures outlined later in this chapter. In order for you to fully understand the purpose of these checks, the use of the following diagnostic tools is explained here.

Purge Flow Controller/Pressure Gauge

The 4560 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. A pressure gauge is connected to the output of the Flow Controller so the gas pressure required to maintain the set flow can be monitored. In PURGE, the purge gas flows sequentially through:

- the Purge Valve
- the Purge Vessel
- the Valve Oven Lines
- the Trap
- the Bake Valve to vent

The total restriction caused by these components generates a backpressure of about 5-8 psi in order to maintain 40 ml/min purge flow. If a pressure above about 10 psi is observed during PURGE, one of these five components is causing too great a restriction. 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 and the seal at the sparger neck are the best place to look first. To aid in locating a leak of this type, plug the barbed fitting where purge flow is measured. 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	18 psi
• PURGE COMPLETE/DESORB READY	20 psi
• DESORB W/DRAIN	0 psi
• BACKFLUSH BAKE (W/O PURGE)	20 psi
• BACKFLUSH BAKE (W/ PURGE)	2-3 psi
• DESORB W/O DRAIN	20 psi

Purge gas is not flowing during states in which the gauge reads 20 psi. 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 preset flow. This supply pressure is called the "system pressure" and is regulated to 20 psi by the internal pressure regulator.

The trap is not in line with the purge gas during states in which the gauge reads 2-3 psi, so less pressure is required to maintain the preset 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.

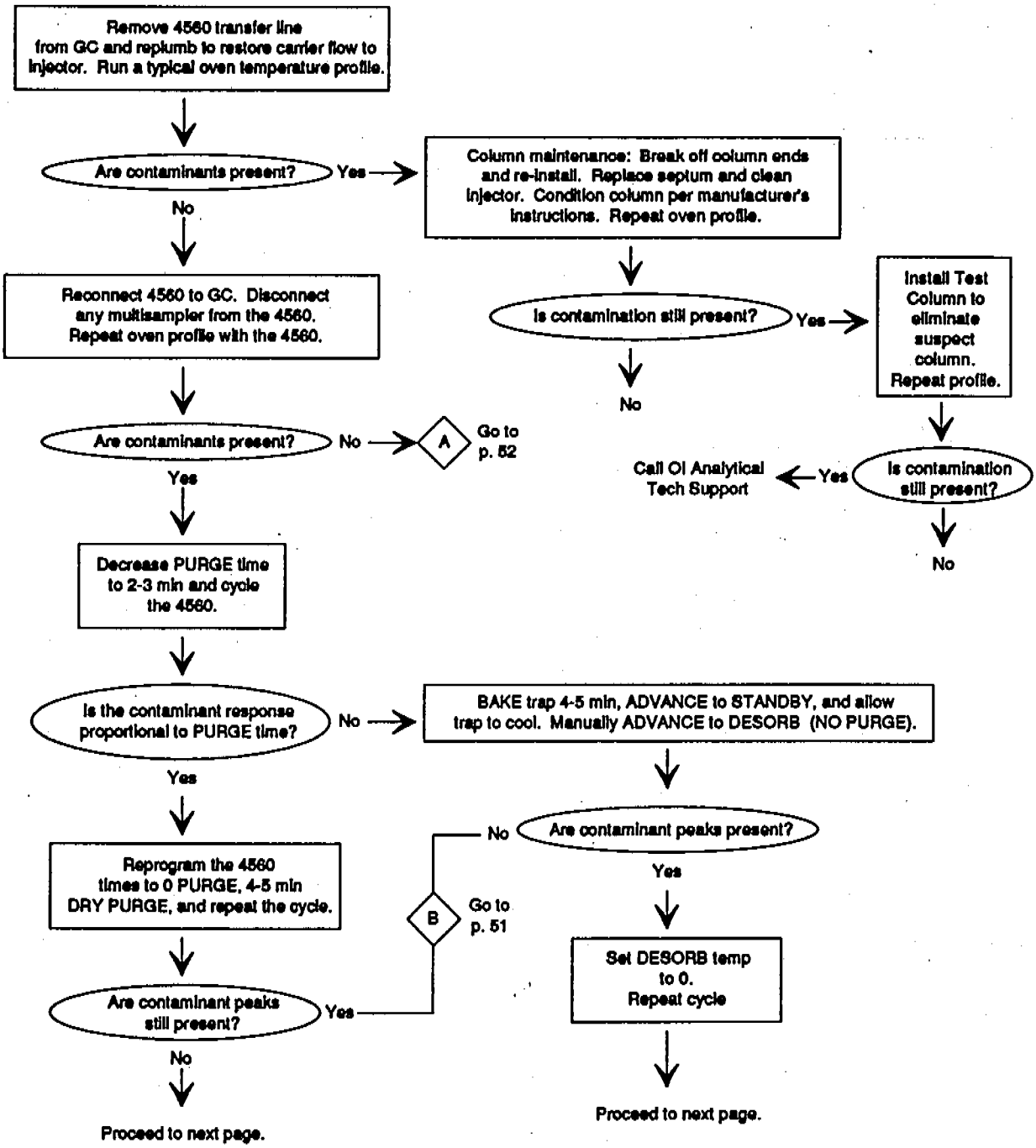
Carrier Flow Controller/Pressure Gauge

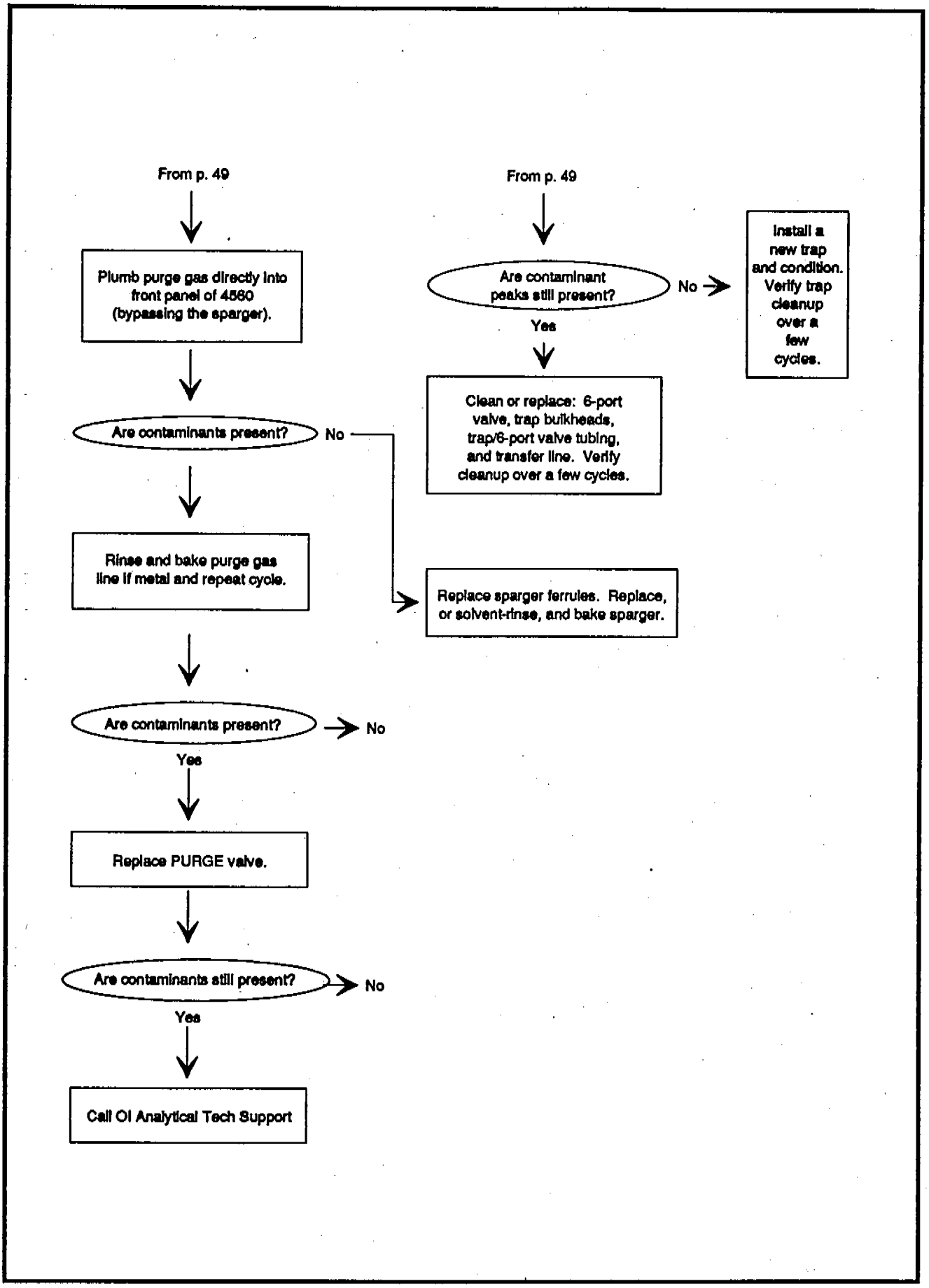
Built into your GC (with Internal Carrier Flow Option) or 4560 is a Flow Controller for regulating the flow of carrier gas through the Column. If this Flow Controller has a pressure gauge connected to its outlet to measure Column head pressure, then it can be used to isolate any loss of response or change 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 I.D. 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 4560 has been connected to the GC, the flow from the Controller goes through the 4560 Trap Valve, back out the heated Transfer Line, and into the GC Injector and Column. Once the system is properly connected, 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 Flow Controller may be unsuitable for flow rates less than 10-20 ml/min.

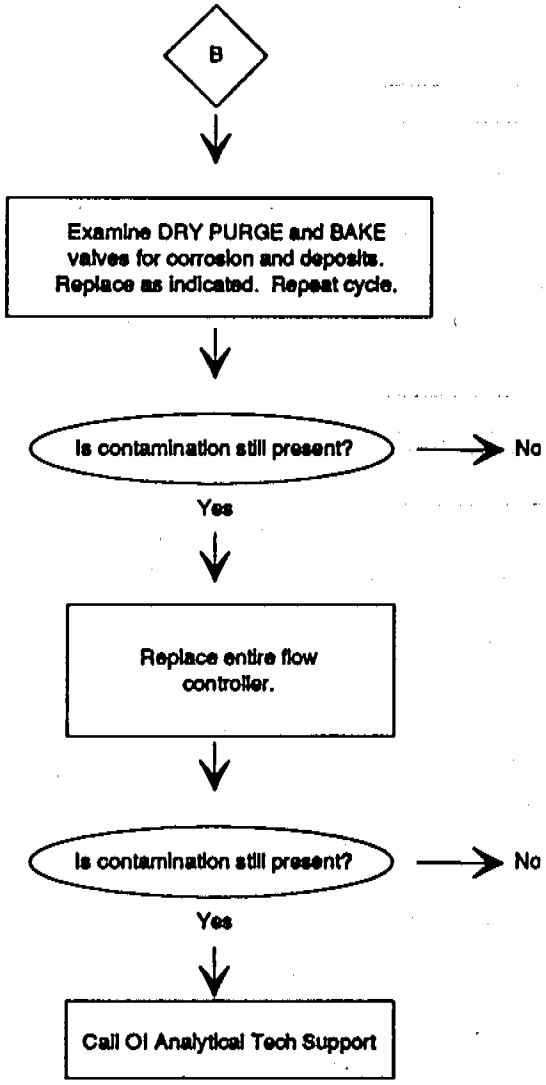
Troubleshooting

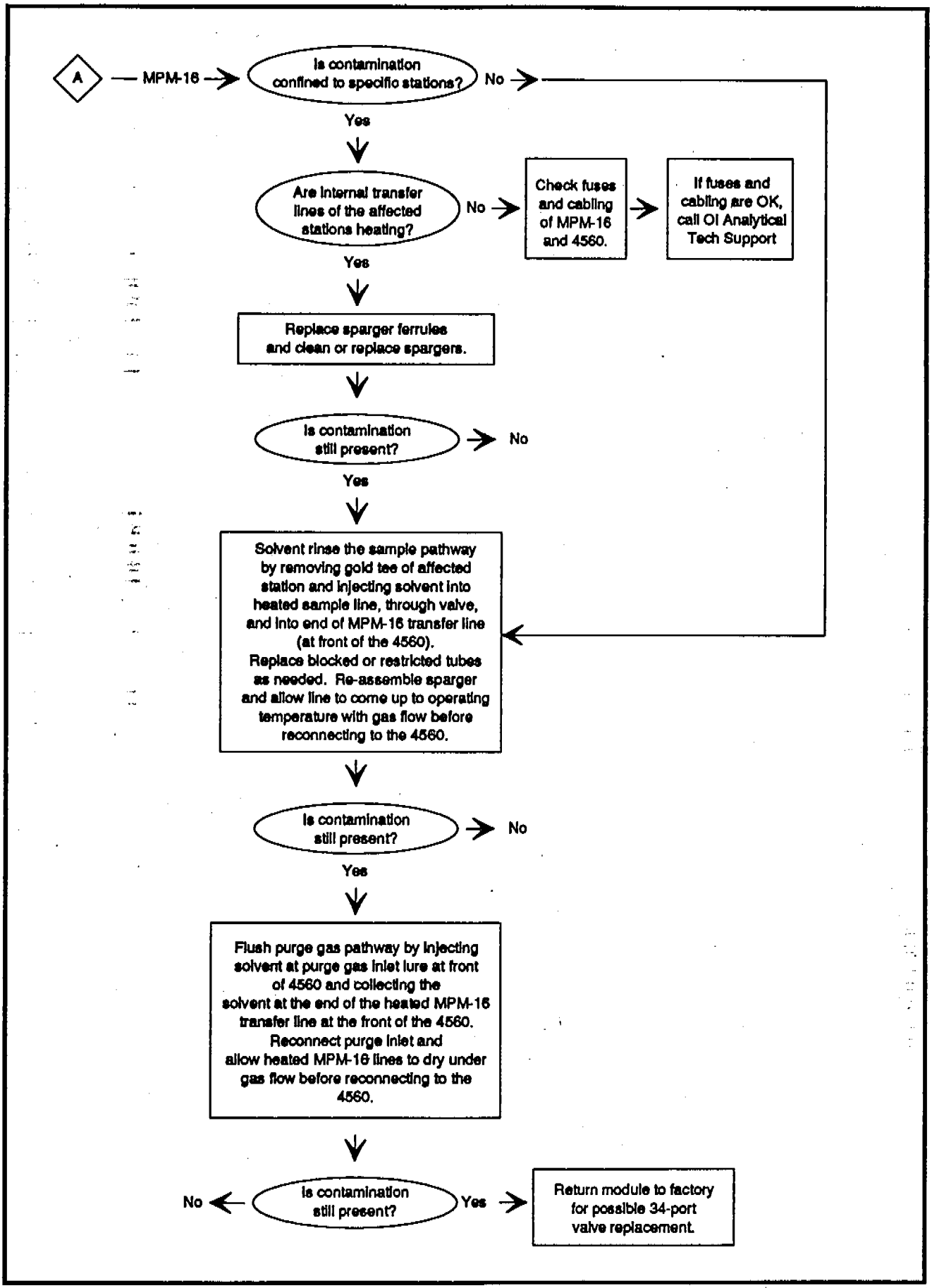
To troubleshoot the 4560, follow the tree diagrams on the following pages for your specific symptoms. If you have multiple symptoms, you may want to choose the most prominent and follow suggestions for that symptom. If the 4560 fails to pass a check, diagnose and correct the problem based on the above discussions. If the system passes these checks, the fault is not likely to be with the 4560.

Contaminant Peaks

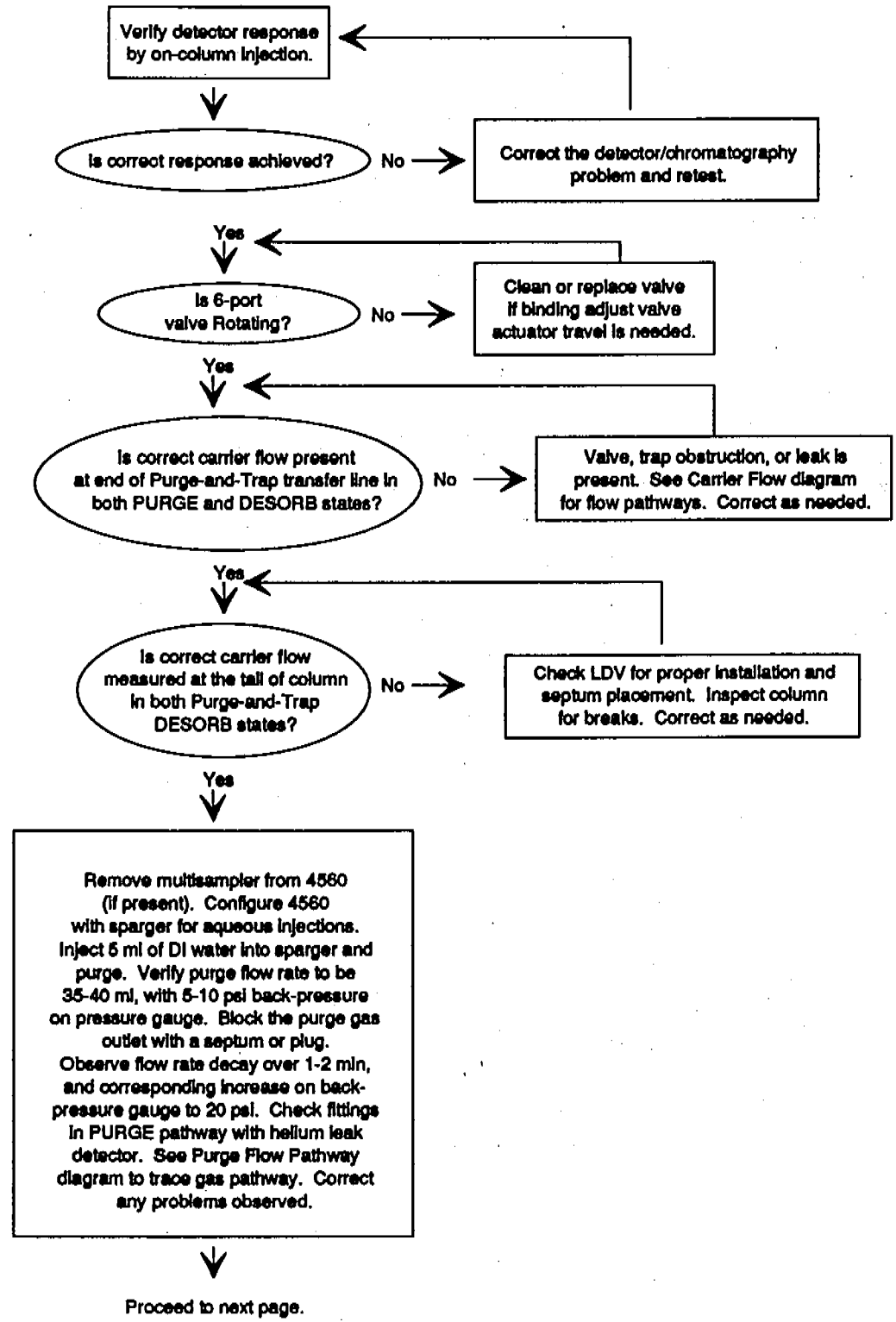


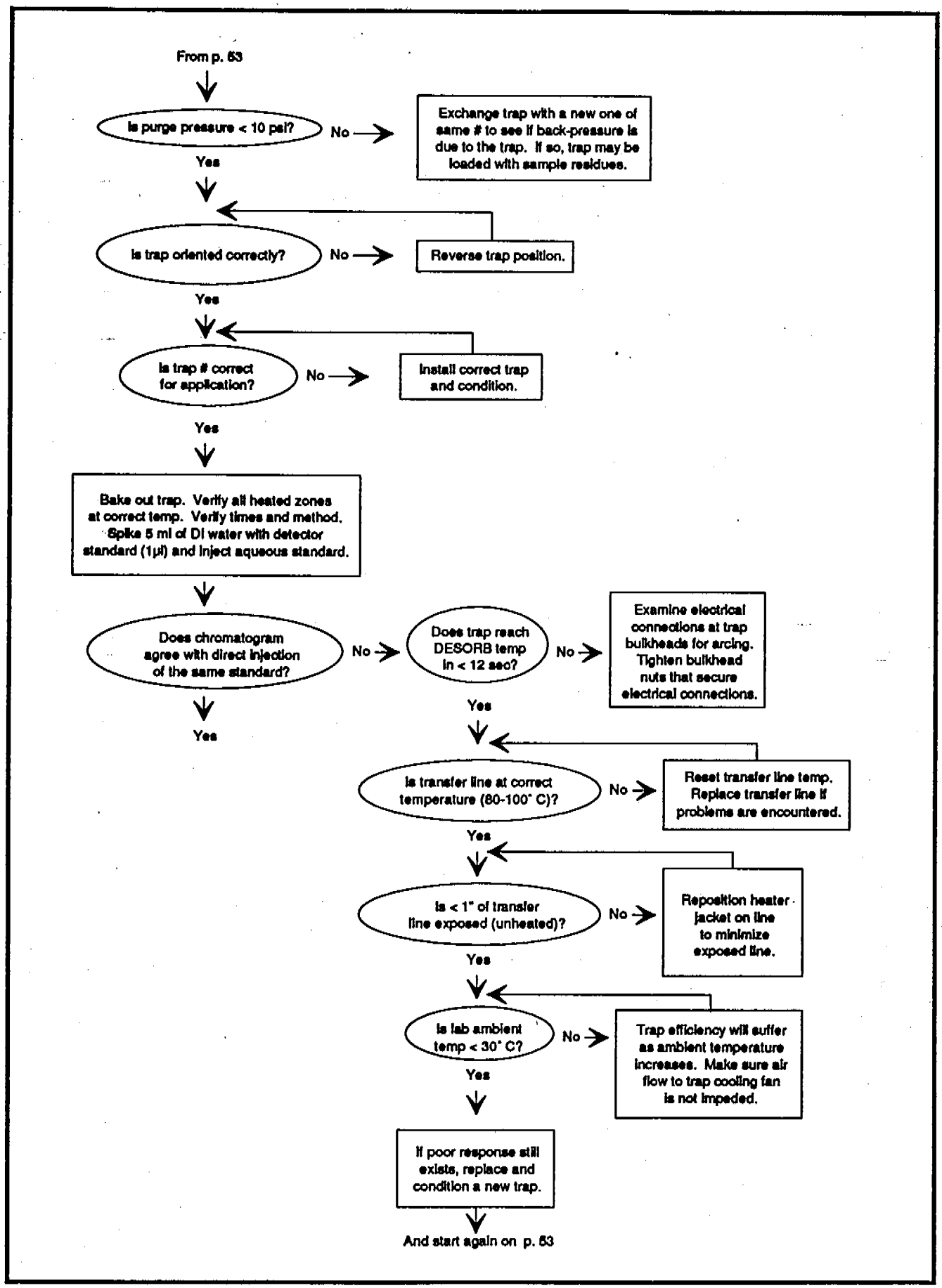

From p. 49
Plumb purge gas directly into front panel of 4560 (bypassing the sparger).
Are contaminants present?
No
Rinse and bake purge gas line if metal and repeat cycle.
Are contaminants present?
No
Yes
Replace PURGE valve.
Are contaminants still present?
No
Yes
Call OI Analytical Tech Support
From p. 49
Are contaminant peaks still present?
No
Install a new trap and condition. Verify trap cleanup over a few cycles.
Yes
Clean or replace: 6-port valve, trap bulkheads, trap/6-port valve tubing, and transfer line. Verify cleanup over a few cycles.
Replace sparger ferrules. Replace, or solvent-rinse, and bake sparger.


B
Examine DRY PURGE and BAKE valves for corrosion and deposits. Replace as indicated. Repeat cycle.
Is contamination still present?
No
Yes
Replace entire flow controller.
Is contamination still present?
No
Yes
Call OI Analytical Tech Support

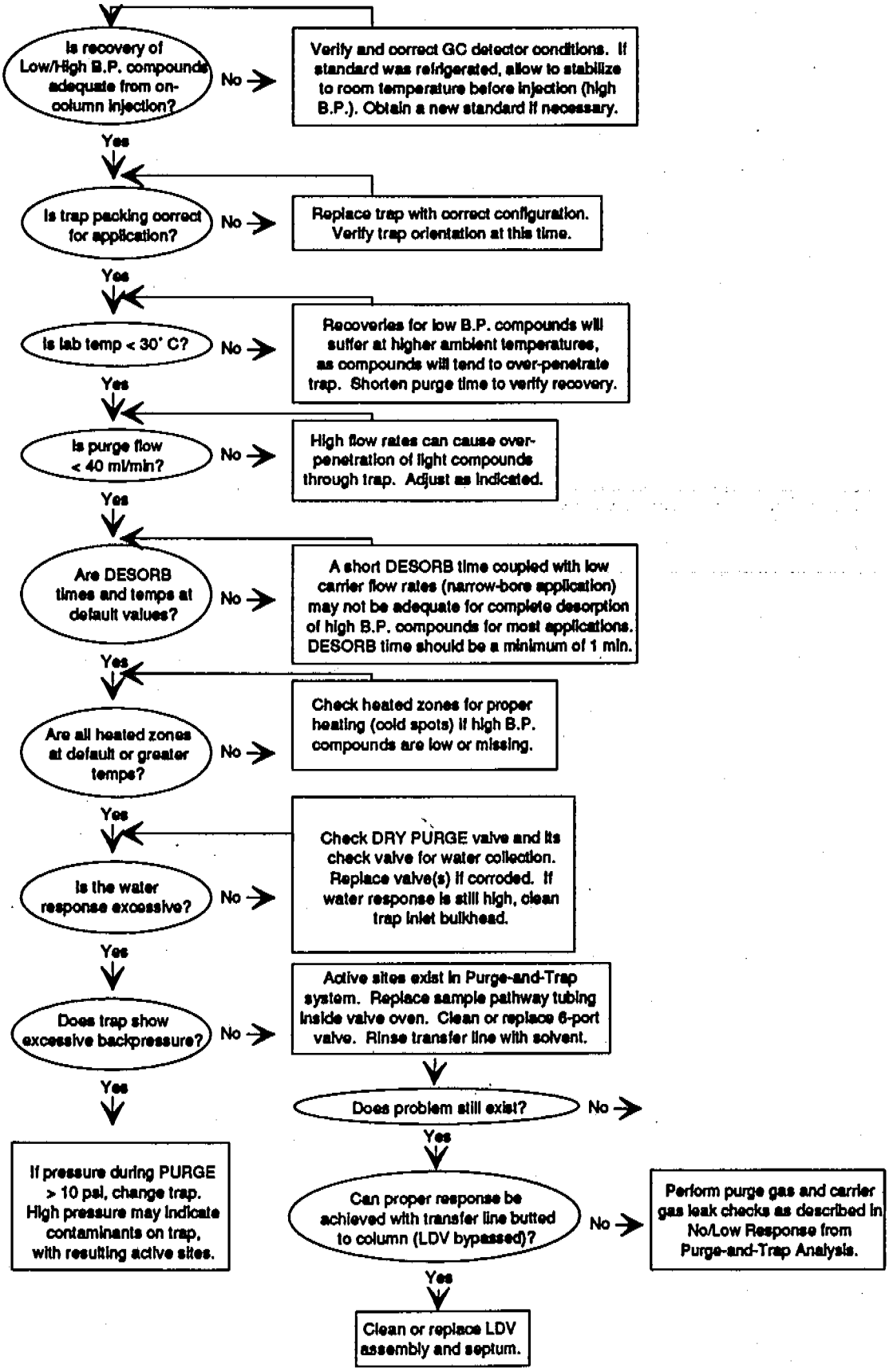

A
MPM-16
Is contamination confined to specific stations?
No
Yes
Are internal transfer lines of the affected stations heating?
No
Check fuses and cabling of MPM-16 and 4560.
If fuses and cabling are OK, call OI Analytical Tech Support
Yes
Replace sparger ferrules and clean or replace spargers.
Is contamination still present?
No
Yes
Solvent rinse the sample pathway by removing gold tee of affected station and injecting solvent into heated sample line, through valve, and into end of MPM-16 transfer line (at front of the 4560). Replace blocked or restricted tubes as needed. Re-assemble sparger and allow line to come up to operating temperature with gas flow before reconnecting to the 4560.
Is contamination still present?
No
Yes
Flush purge gas pathway by injecting solvent at purge gas inlet lure at front of 4560 and collecting the solvent at the end of the heated MPM-16 transfer line at the front of the 4560. Reconnect purge inlet and allow heated MPM-16 lines to dry under gas flow before reconnecting to the 4560.
Is contamination still present?
No
Yes
Return module to factory for possible 34-port valve replacement.

No/Low Response from Purge-and-Trap Analysis

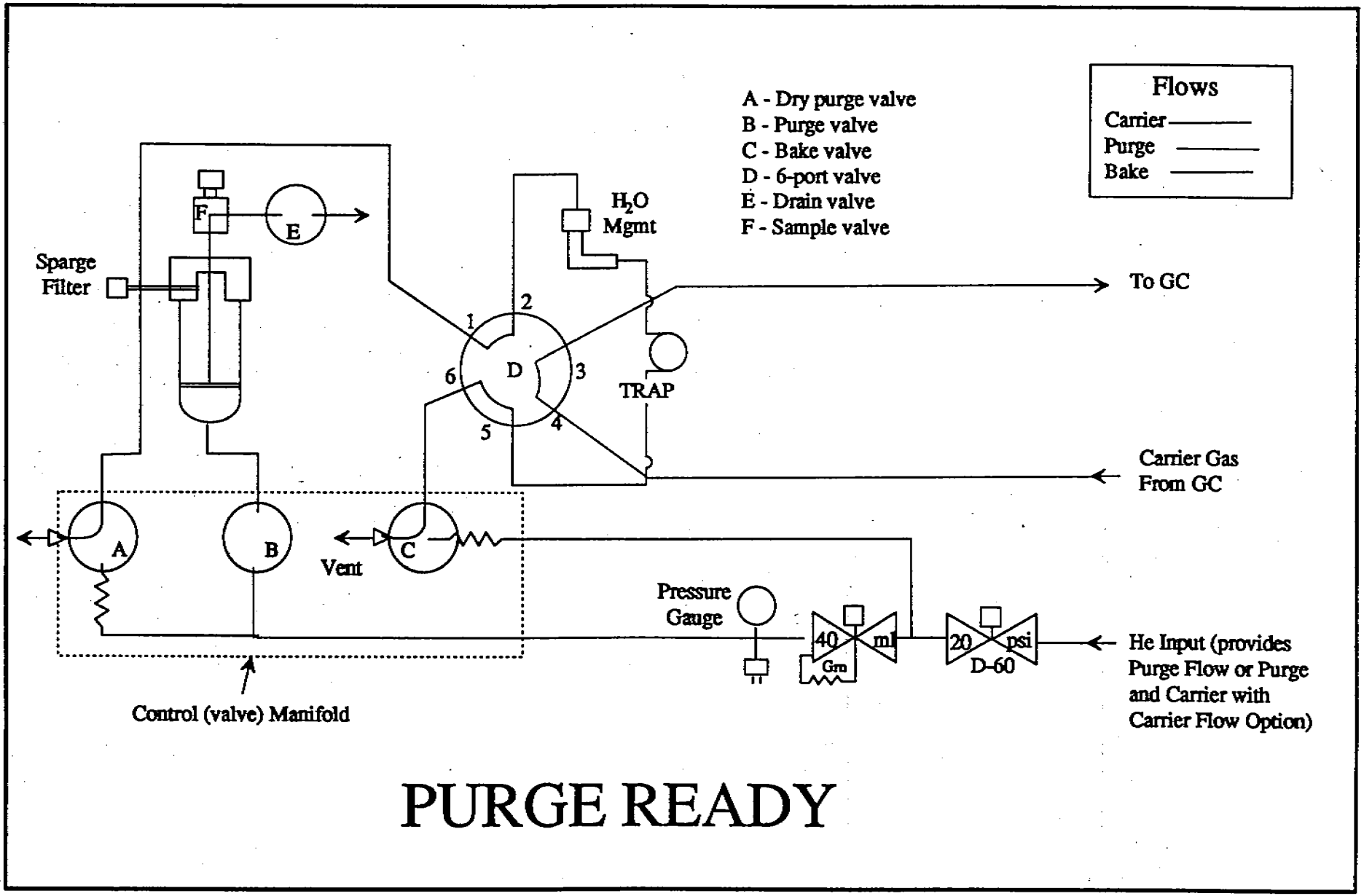


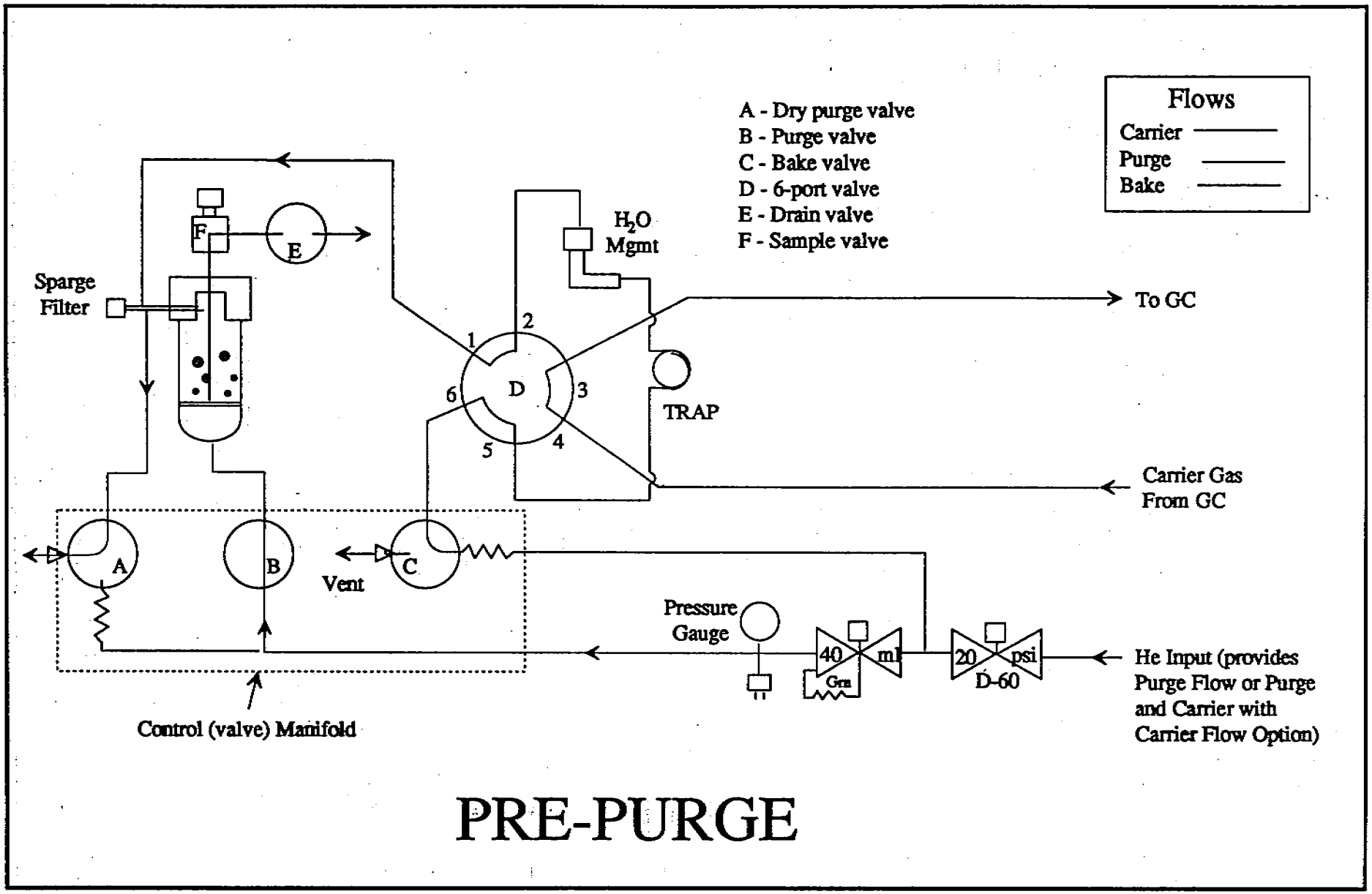

From p. 53
Is purge pressure < 10 psi?
No
Exchange trap with a new one of same # to see if back-pressure is due to the trap. If so, trap may be loaded with sample residues.
Yes
Is trap oriented correctly?
No
Reverse trap position.
Yes
Is trap # correct for application?
No
Install correct trap and condition.
Yes
Bake out trap. Verify all heated zones at correct temp. Verify times and method. Spike 5 ml of DI water with detector standard (1µl) and inject aqueous standard.
Does chromatogram agree with direct injection of the same standard?
No
Does trap reach DESORB temp in < 12 sec?
No
Examine electrical connections at trap bulkheads for arcing. Tighten bulkhead nuts that secure electrical connections.
Yes
Yes
Is transfer line at correct temperature (80-100° C)?
No
Reset transfer line temp. Replace transfer line if problems are encountered.
Yes
Is < 1" of transfer line exposed (unheated)?
No
Reposition heater jacket on line to minimize exposed line.
Yes
Is lab ambient temp < 30° C?
No
Trap efficiency will suffer as ambient temperature increases. Make sure air flow to trap cooling fan is not impeded.
Yes
If poor response still exists, replace and condition a new trap.
And start again on p. 53

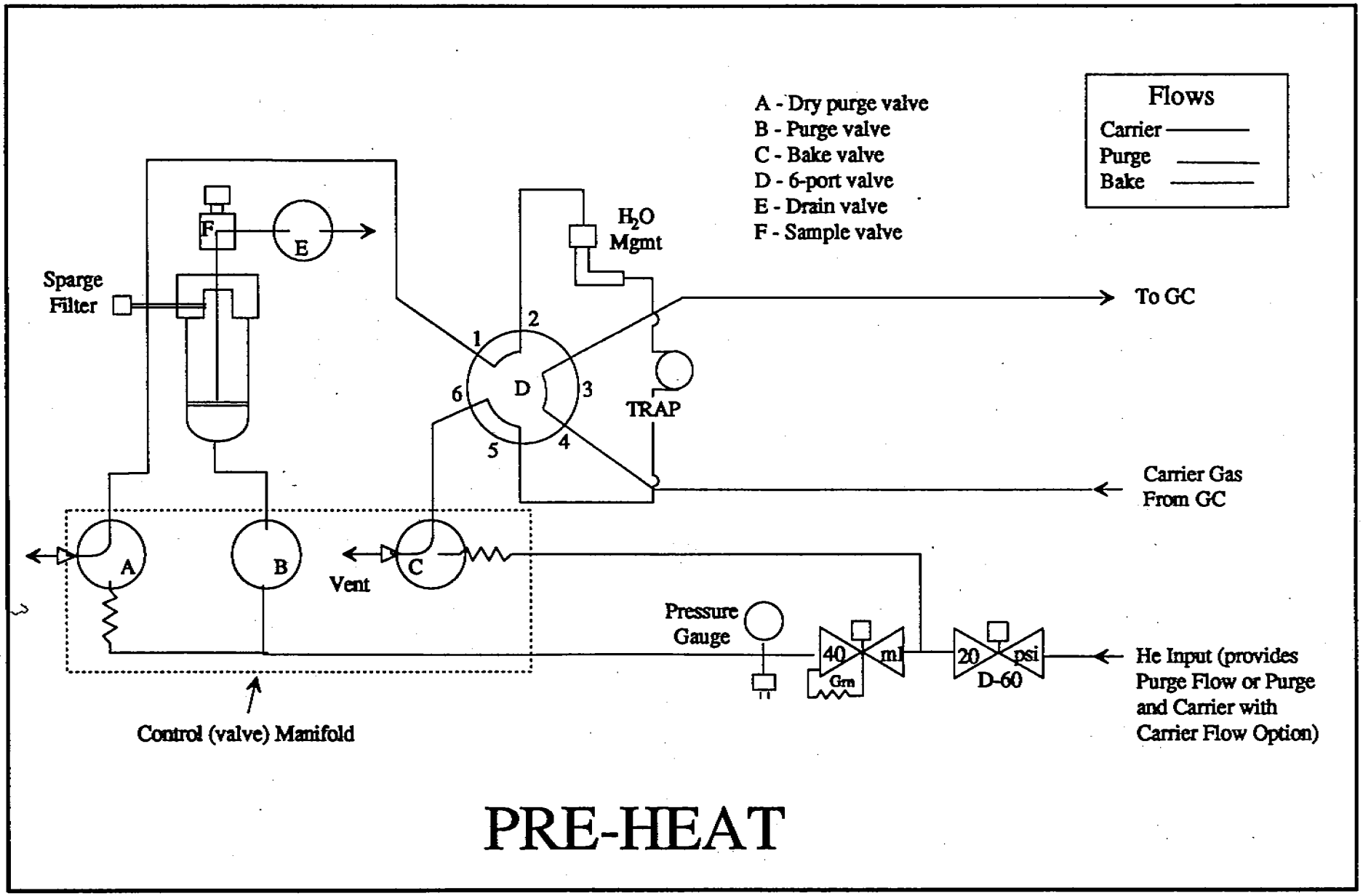
Poor Recovery of Low/High Boiling Point Compounds

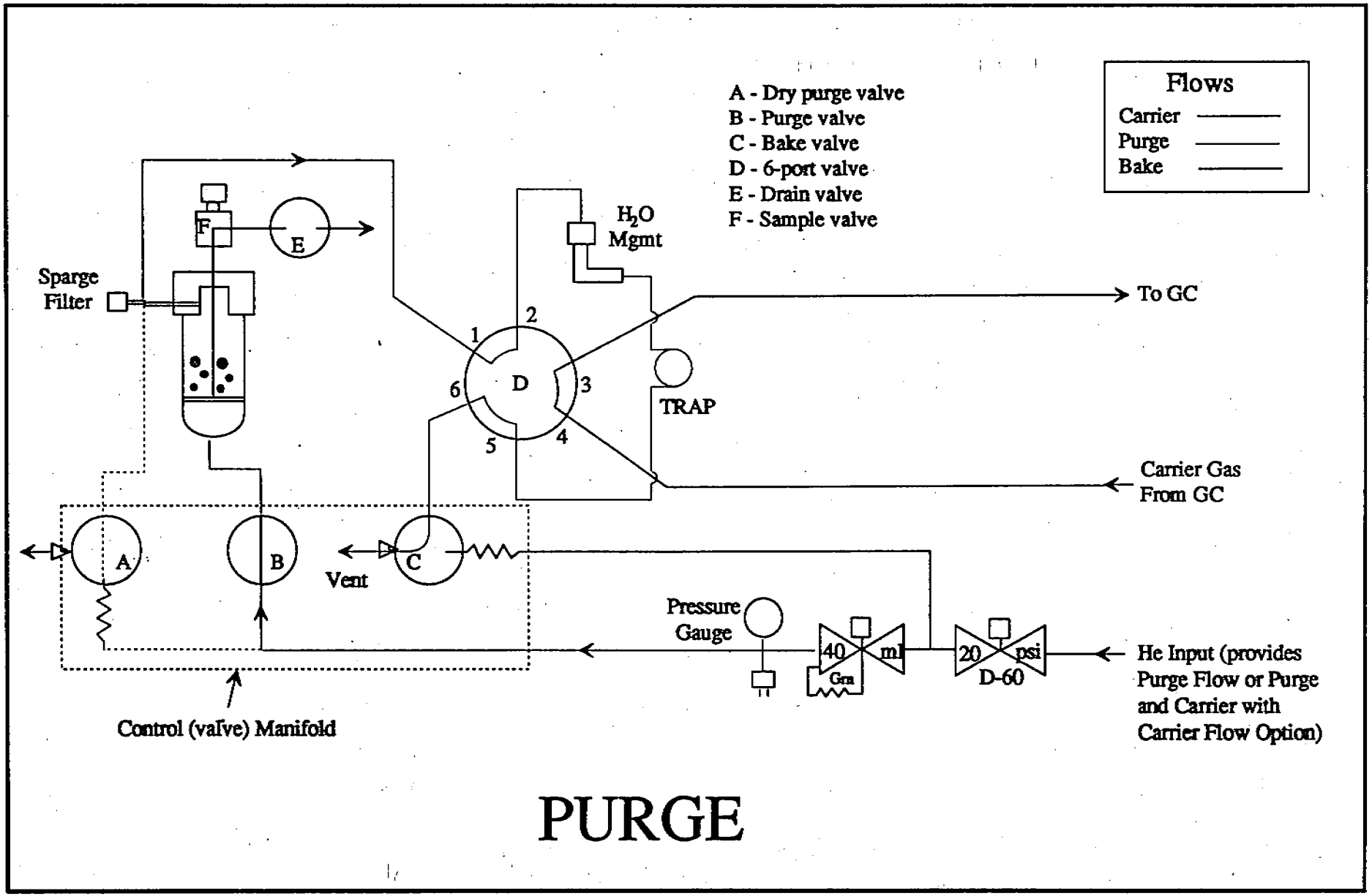


NOTES

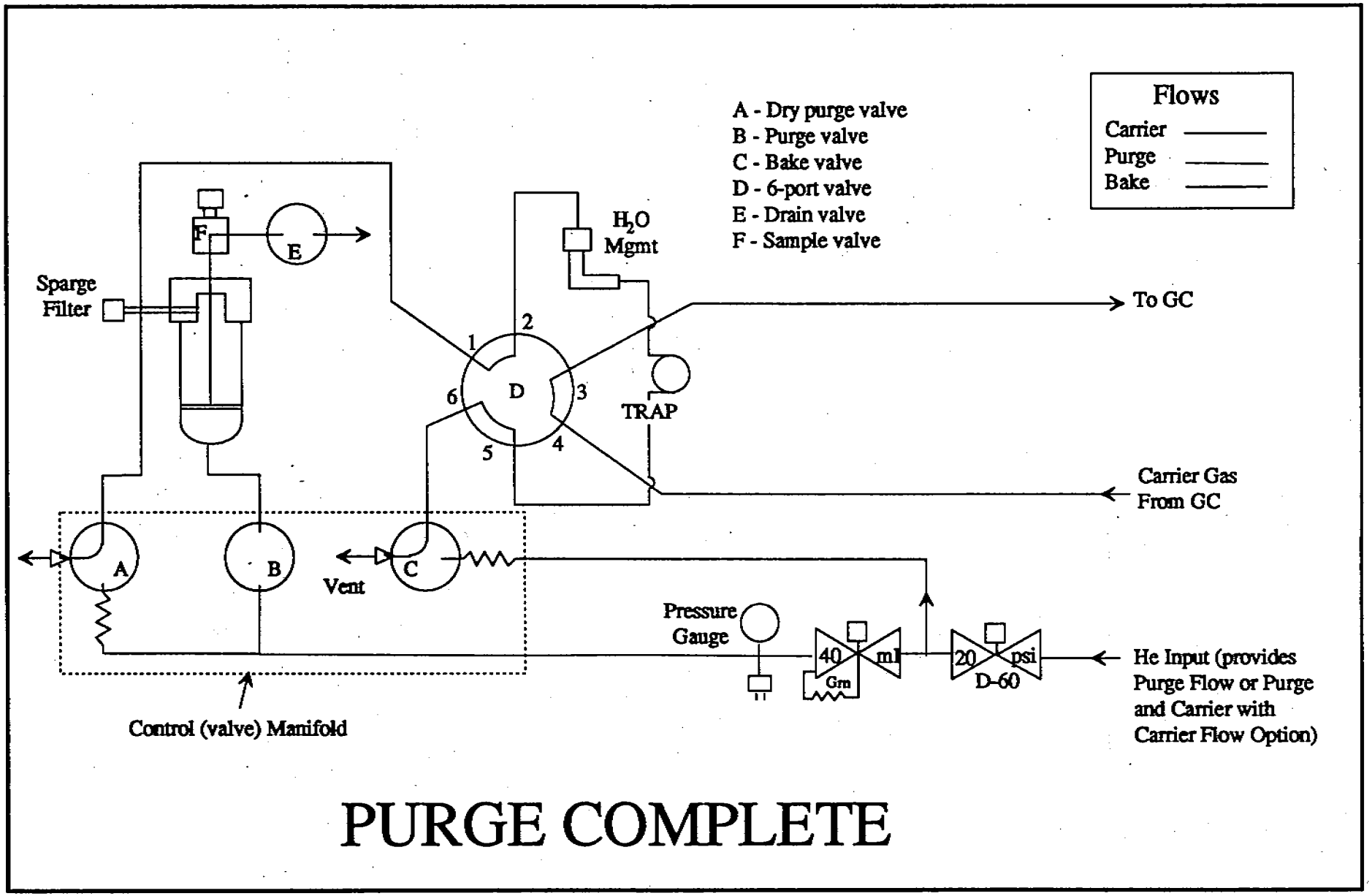

A - Dry purge valve
B - Purge valve
C - Bake valve
D - 6-port valve
E - Drain valve
F - Sample valve
Flows
Carrier
Purge
Bake
H_2O Mgmt
Sparge Filter
To GC
TRAP
Carrier Gas From GC
Vent
Pressure Gauge
40 ml Grn
20 psi D-60
He Input (provides Purge Flow or Purge and Carrier with Carrier Flow Option)
Control (valve) Manifold
PURGE READY

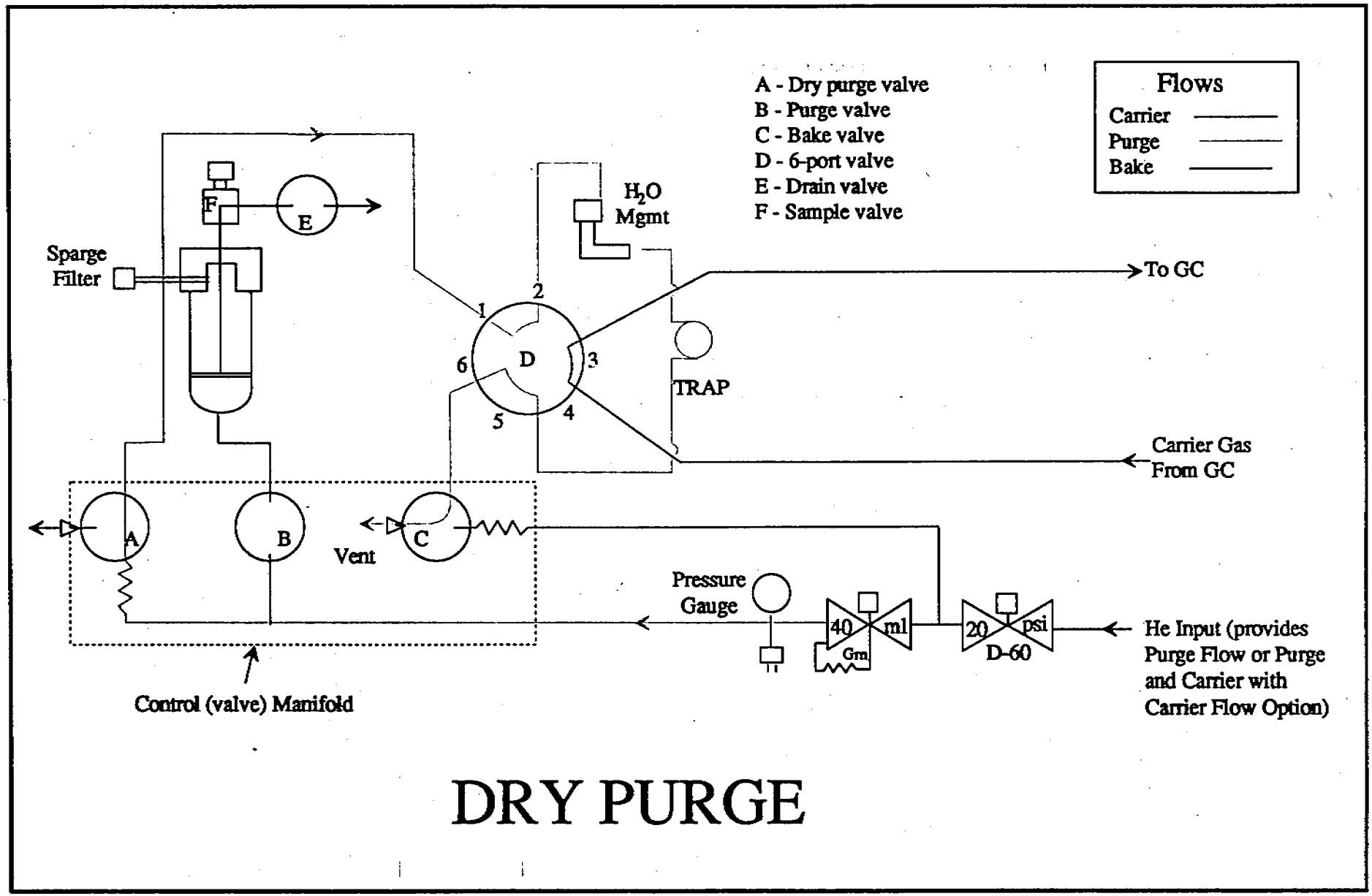

A - Dry purge valve
B - Purge valve
C - Bake valve
D - 6-port valve
E - Drain valve
F - Sample valve
Flows
Carrier
Purge
Bake
H_2O Mgmt
Sparge Filter
To GC
TRAP
Carrier Gas From GC
Vent
Pressure Gauge
40 ml Grn
20 psi D-60
He Input (provides Purge Flow or Purge and Carrier with Carrier Flow Option)
Control (valve) Manifold
PRE-PURGE


A - Dry purge valve
B - Purge valve
C - Bake valve
D - 6-port valve
E - Drain valve
F - Sample valve
Flows
Carrier
Purge
Bake
H_2O Mgmt
Sparge Filter
To GC
TRAP
Carrier Gas From GC
Vent
Pressure Gauge
40 ml Grn
20 psi D-60
He Input (provides Purge Flow or Purge and Carrier with Carrier Flow Option)
Control (valve) Manifold
PRE-HEAT


A - Dry purge valve
B - Purge valve
C - Bake valve
D - 6-port valve
E - Drain valve
F - Sample valve
Flows
Carrier
Purge
Bake
F
E
H_2O Mgmt
Sparge Filter
To GC
1
2
3
4
5
6
D
TRAP
Carrier Gas From GC
A
B
C
Vent
Pressure Gauge
40 ml Gas
20 psi D-60
He Input (provides Purge Flow or Purge and Carrier with Carrier Flow Option)
Control (valve) Manifold

PURGE


A - Dry purge valve
B - Purge valve
C - Bake valve
D - 6-port valve
E - Drain valve
F - Sample valve
Flows
Carrier
Purge
Bake
H_2O Mgmt
Sparge Filter
To GC
TRAP
Carrier Gas From GC
Vent
Pressure Gauge
40 ml Gm
20 psi D-60
He Input (provides Purge Flow or Purge and Carrier with Carrier Flow Option)
Control (valve) Manifold
PURGE COMPLETE


A - Dry purge valve
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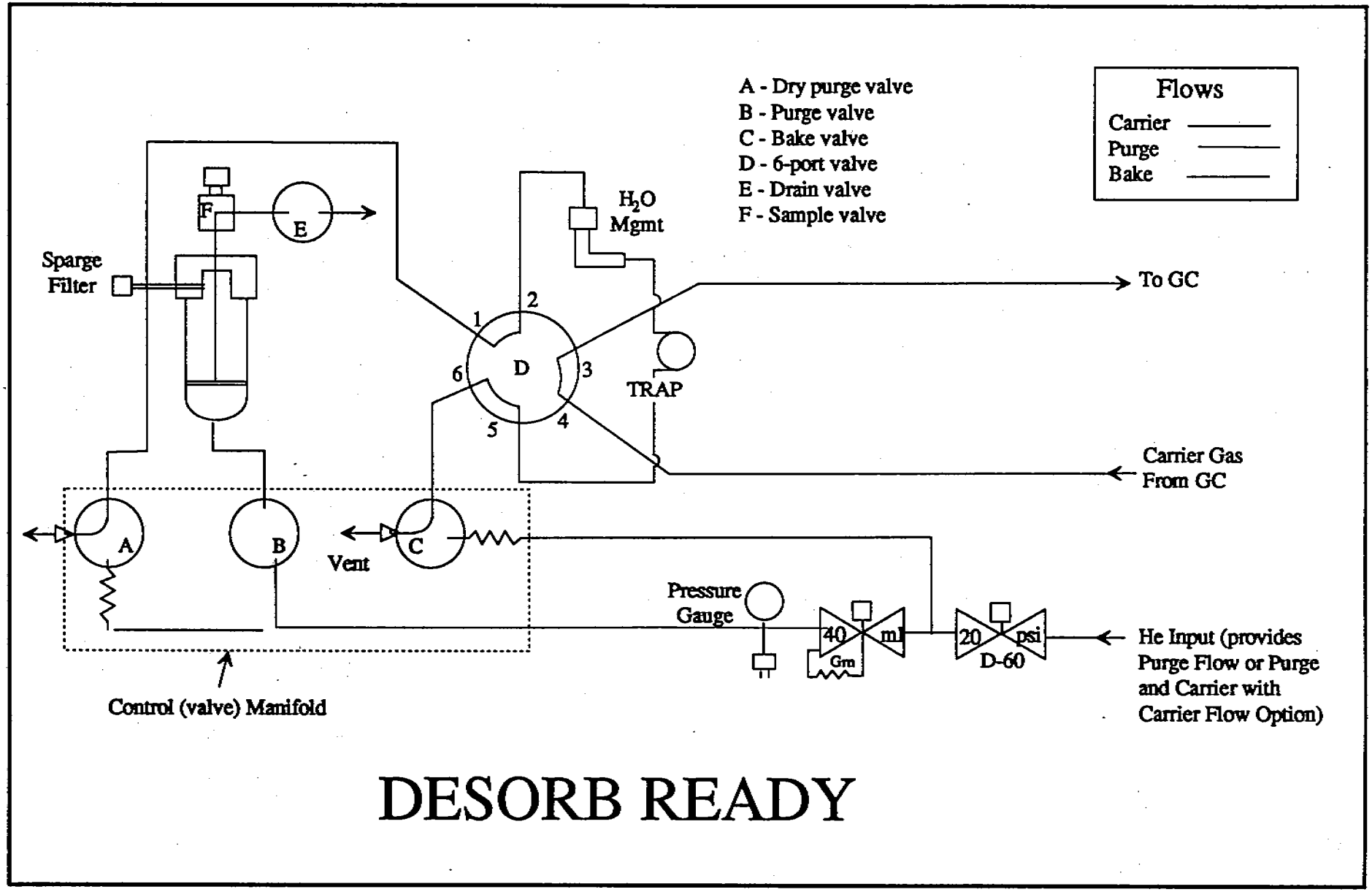
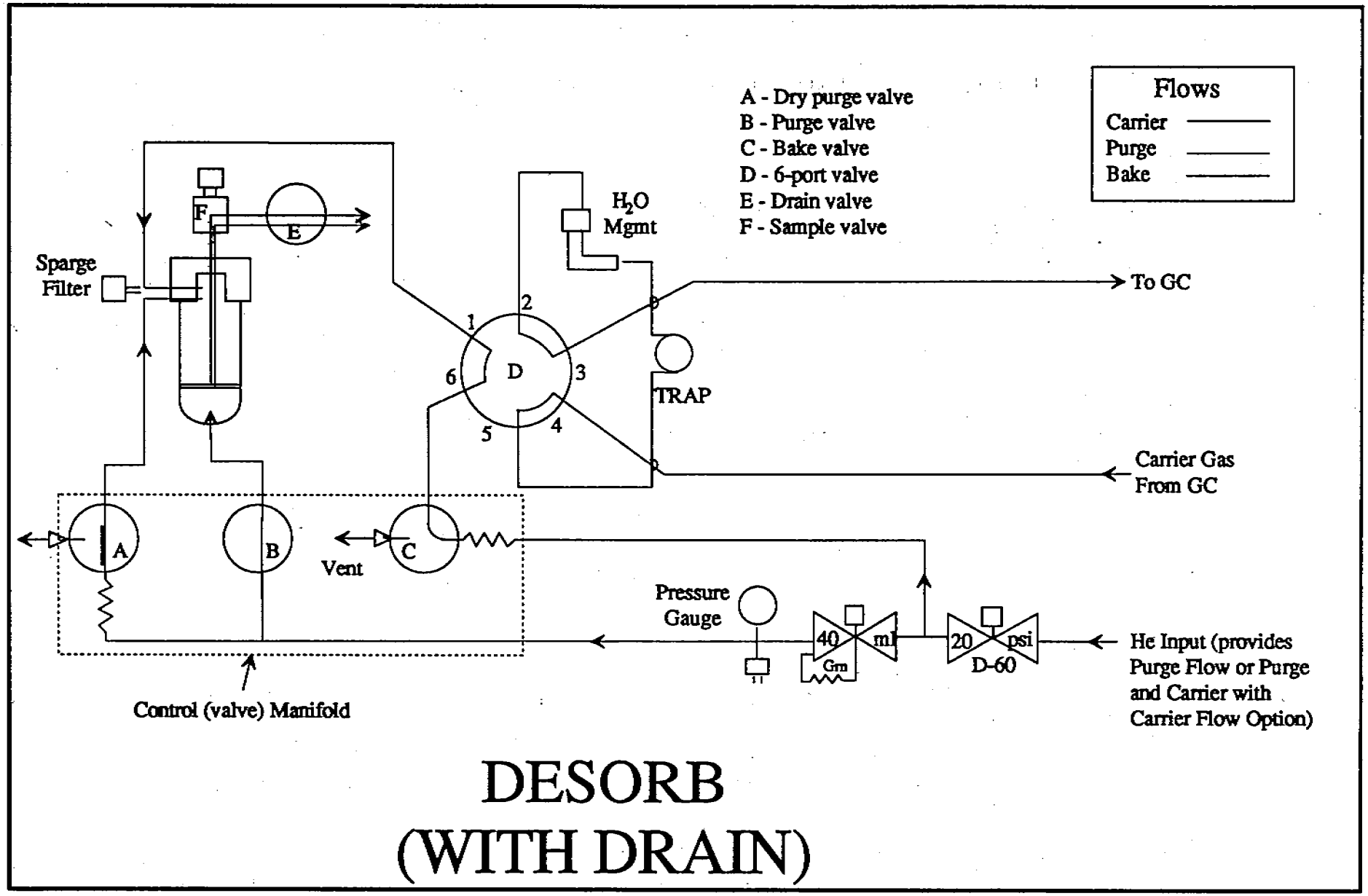
C - Bake valve
D - 6-port valve
E - Drain valve
F - Sample valve
Flows
Carrier
Purge
Bake
F
E
H_2O Mgmt
Sparge Filter
To GC
1
2
3
4
5
6
D
TRAP
Carrier Gas From GC
A
B
C
Vent
Pressure Gauge
40
ml
Gm
20
psi
D-60
He Input (provides Purge Flow or Purge and Carrier with Carrier Flow Option)
Control (valve) Manifold

DRY PURGE



DESORB READY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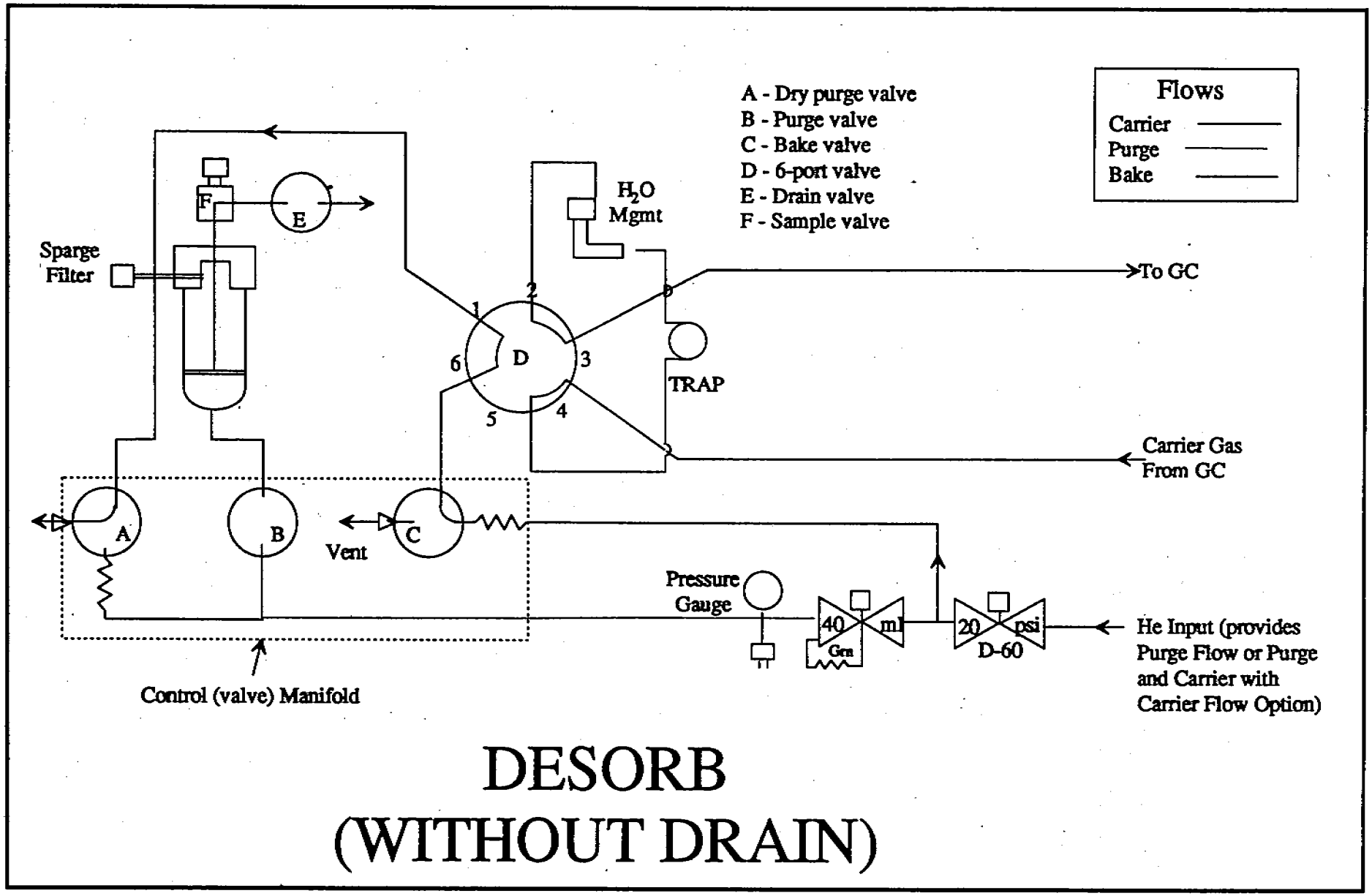
C - Bake valve
D - 6-port valve
E - Drain valve
F - Sample valve
Flows
Carrier
Purge
Bake
H_2O Mgmt
Sparge Filter
To GC
TRAP
Carrier Gas From GC
Vent
Pressure Gauge
40 ml
Gm
20 psi
D-60
He Input (provides Purge Flow or Purge and Carrier with Carrier Flow Option)
Control (valve) Manifold
DESORB
(WITH DRAIN)


A - Dry purge valve
B - Purge valve
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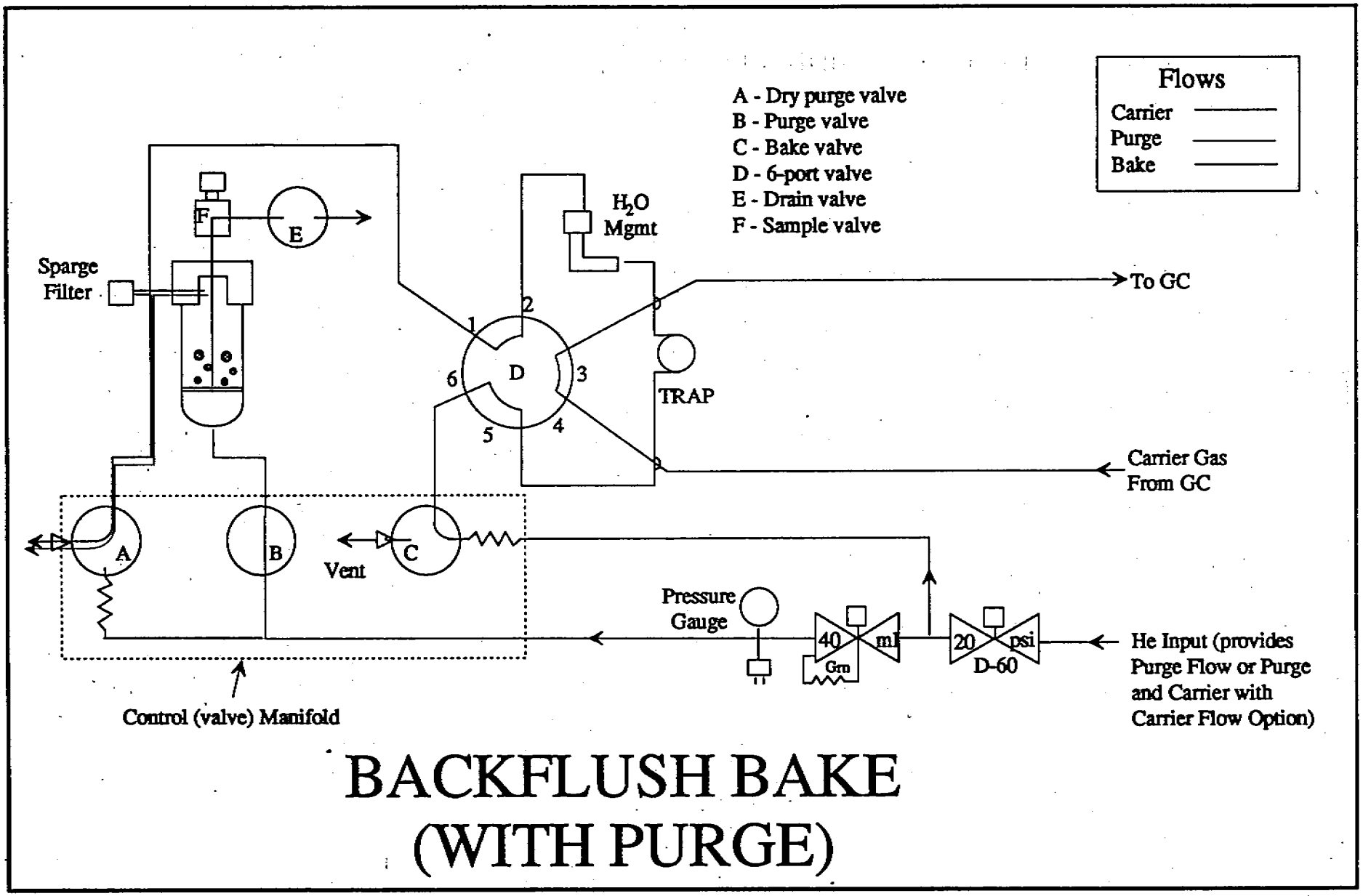
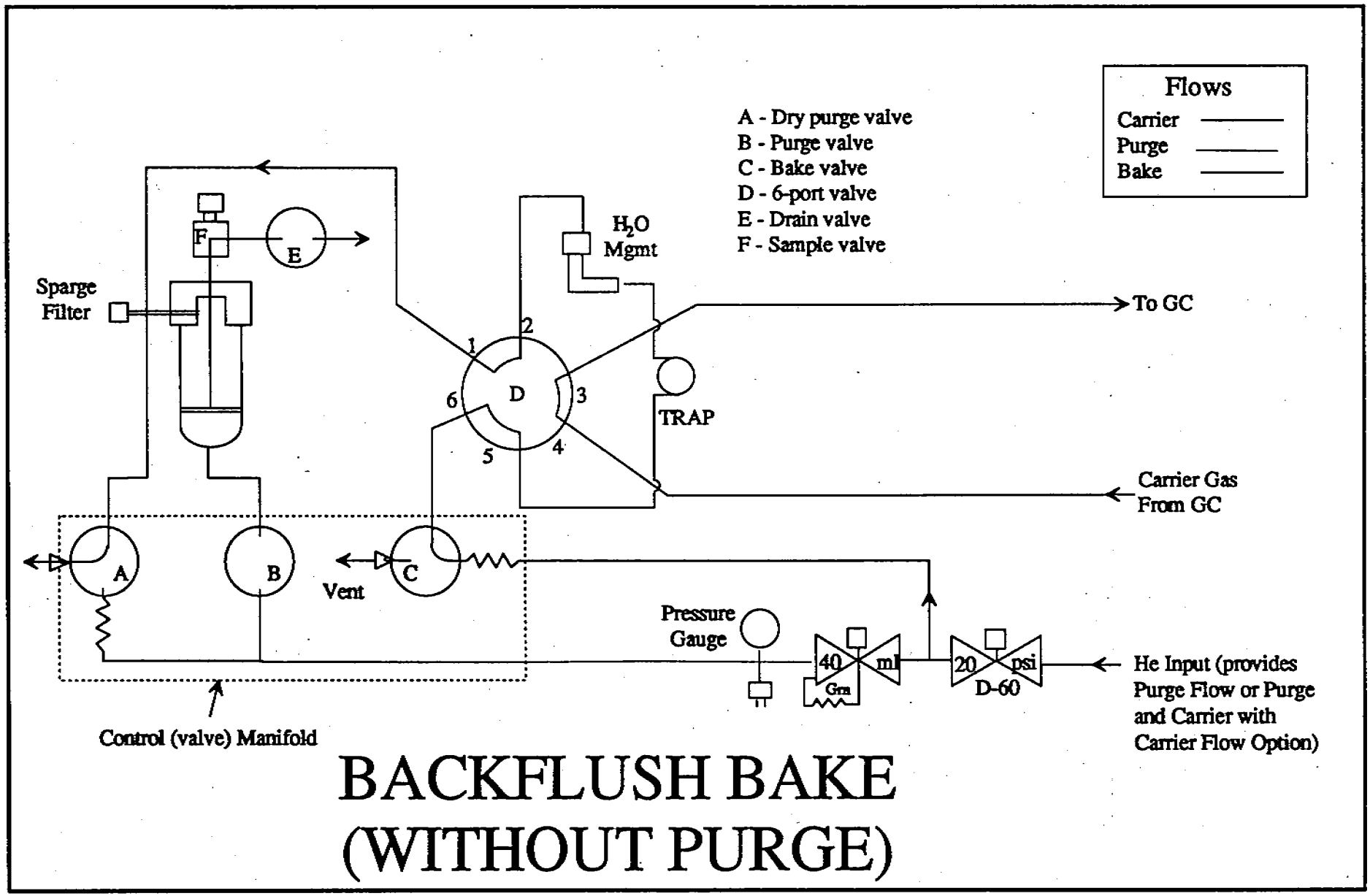
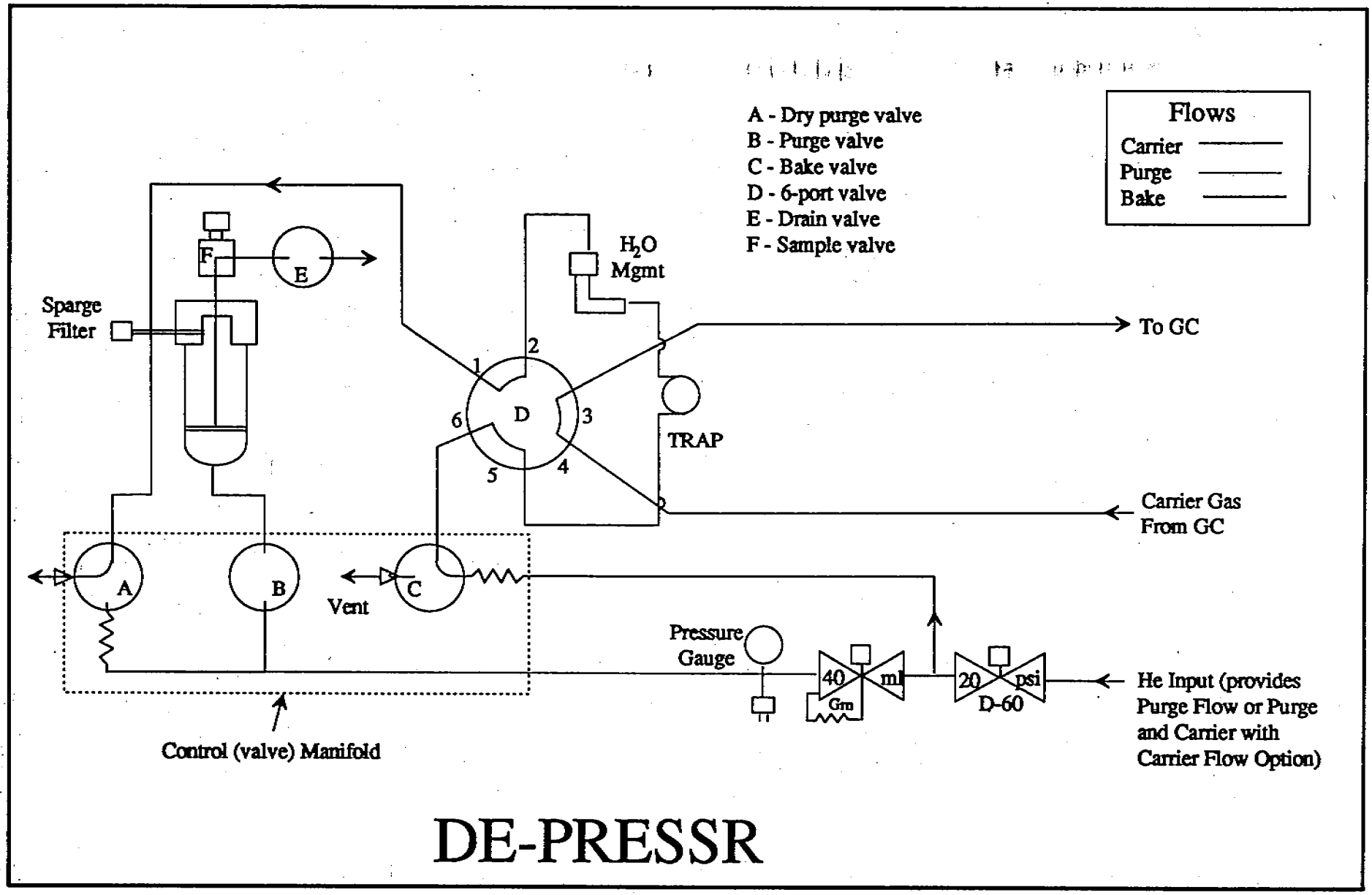
D - 6-port valve
E - Drain valve
F - Sample valve
Flows
Carrier
Purge
Bake
H_2O Mgmt
Sparge Filter
To GC
TRAP
Carrier Gas From GC
Vent
Pressure Gauge
40 ml
Grn
20 psi
D-60
He Input (provides Purge Flow or Purge and Carrier with Carrier Flow Option)
Control (valve) Manifold
DESORB
(WITHOUT DRAIN)


A - Dry purge valve
B - Purge valve
C - Bake valve
D - 6-port valve
E - Drain valve
F - Sample valve
Flows
Carrier
Purge
Bake
H_2O Mgmt
Sparge Filter
To GC
TRAP
Carrier Gas From GC
Vent
Pressure Gauge
40 mL Grn
20 psi D-60
He Input (provides Purge Flow or Purge and Carrier with Carrier Flow Option)
Control (valve) Manifold
BACKFLUSH BAKE (WITH PURGE)



BACKFLUSH BAKE (WITHOUT PURGE)


A - Dry purge valve
B - Purge valve
C - Bake valve
D - 6-port valve
E - Drain valve
F - Sample valve
Flows
Carrier
Purge
Bake
F
E
Sparge Filter
H2O Mgmt
To GC
1
2
3
4
5
6
D
TRAP
Carrier Gas From GC
A
B
C
Vent
Pressure Gauge
40
ml
Gm
20
psi
D-60
He Input (provides Purge Flow or Purge and Carrier with Carrier Flow Option)
Control (valve) Manifold

DE-PRESSR

Chapter 7
Replacement Parts

In Chapter 2, the various components of the Model 4560 were identified and described. This chapter lists the order numbers for replacement parts and support items for the Sample Concentrator and its associated options. Replacement parts considered as expendable (XPND) are marked with an asterisk. A supply of XPND parts should be kept on hand. Units of measure (U/M) are also given.

Parts for 4560 Sample Concentrator

Part Name	Part #	U/M	XPND
Boards			
AC - I/O Board	226993	ea	
Power Supply Board	223396	ea	
Back Plane Board	226910	ea	
4560 CPU Board	228098	ea	
Panel - Control Front 4560	224618	ea	
Cables (Internal to 4560)			
Fan Solenoid	207795	ea	
AC - I/O Board to Pwr Supply (Ribbon)	225284	ea	
Ribbon AC - I/O / CPU	225292	ea	
Display CPU	225300	ea	
Electronic Valve Actuator (EVA)	227025	ea	
EVA to AC - I/O Board	227058	ea	
Power Switch	227066	ea	
Trap Door Switch / AC - I/O Board	227124	ea	
AC - I/O Board / Pwr Supply (4-wire)	227132	ea	
Valve Heater / Oven Heater	227157	ea	
Communication Cables			
Remote Start Pigtail HP 5890	185868	ea	
Handshake - 4560 / HP 5890 Pigtail	226530	ea	
4560 / MHC-16 or MPM-16	226605	ea	
4560 / CFM	226548	ea	
4560 / PATTI®II (RS-232)	226621	ea	
MPM-16 / MHC-16 (Rev B)	229450	ea	

Part Name	Part #	U/M	XPND
MSD Interface Cables (Replace #185868 when MSD present)			
Handshake - Pigtail HP 5971 (UNIX / DOS)	209874	ea	
Handshake - Pigtail HP 5971 (Pascal)	209866	ea	
Handshake - Pigtail HP 5970 (RTEA)	209882	ea	
Fans			
Fan Assy - Trap 4560	227280	ea	
Fan Assy - Electronics 4560	227322	ea	
Fan Assy - Left Bay (Intake)	227355	ea	
Fan Assy - Water Mgmt 4560	230086	ea	
Fittings and Ferrules			
Ferrule - TFE 1/16 Tube	177626	ea	*
Ftng - Nut SS 1/16 Male Knurl	217240	ea	*
Ferrule - TFE 6 mm Tube (Sparger Inlet Arm)	224337	ea	*
Ferrule - TFE 18 mm Tube (10/pk) (Spgr. Mount)	224352	pk	*
Ftng - Adapt Kel-F F-Luer 10-32 (Syringe Port)	224584	ea	*
Ftng - Union Br 1/4 - 1/16 Male (Spgr. Inlet Arm)	227223	ea	*
Software Control			
PATTI® II with Super Sequencing	232389	ea	
Spargers (Purge Vessels)			
20 ml Disposable (125/box) 18 mm	199521	box	*
5 ml Frit 18 mm	209015	ea	*
25 ml Frit 18 mm	209031	ea	*
5 ml Needle 18 mm	225623	ea	*
25 ml Needle 18 mm	225631	ea	*
Sparger Kits (includes hardware)			
Needle Sparge Hardware Kit	227397	ea	
Traps and Trap Bulkhead Parts			
Spacer - Ftng Bulkhd Trap 4560	206243	ea	*
Washer - Bulkhead Trap (PEEK)	206250	ea	*
Ftng - Union Bulkhead SS/Au 1/8 M-F	209098	ea	
Washer - Water Mgmt PEEK 4560	209478	ea	*
Trap - SS #9 (Tenax / Silica Gel / Charcoal)	219972	ea	*
Trap - SS #7 (Tenax only)	227348	ea	*
Trap - SS #8 (Tenax / Silica Gel)	227363	ea	*
Trap - SS #0 (Blank)	228114	ea	*

Part Name	Part #	U/M	XPND
Tubing and Tube Assemblies			
Tubing - Copper 1/8 x 0.070	111427	ft	
Tubing - Urethane 1/8 x 1/16 Clear (Drain Line)	166224	ft	
Tube Assy - Purge Gas / Needle Sparger	227389	ea	
Tube Assy - Wtr Mgmt / 6-Port Valve	227439	ea	*
Tube Assy - Solenoid Manifold / 6-Port Valve	227447	ea	*
Tube Assy - Regulator Out / Solenoid Manifold	227454	ea	
Tube Assy - Purge Bulkhead / Regulator 4560	227520	ea	
Tube Assy - Tee / 6-Port Valve	227553	ea	
Tube Assy - Carrier FC / Tee	227561	ea	
Tube Assy - Carrier Bulkhead / Tee	227579	ea	
Tube Assy - Purge Out / Gauge 4560	227603	ea	
Tube Assy - Purge FC Out / Solenoid Manifold	227611	ea	
Tube Assy - Solenoid Manifold Jumper	227629	ea	
Tube Assy - Trap / 6-Port Valve	227637	ea	*
Tube Assy - 6-Port Valve / Sparge Manifold	227645	ea	*
Tube Assy - Purge Gas / Frit Sparger	227652	ea	*
Xfer Line - Ni 48 in x 0.020 ID	228064	ea	
Tube Assy - Valve Solenoid / Sparge Manifold	228957	ea	
Other Parts 4560			
Power Cord	116038	ea	
Syringe - 10 ul x 2 in Needle	167545	ea	*
Fuse 8 Amp Slo-Blo	176851	ea	*
Syringe - 25 ml Sample w/ Luer	177659	ea	*
Valve - Br/Ni 3-way Mfld VTN 12 V	179325	ea	
Valve - BR/Ni Check 1/2 psi 10-32	199984	ea	
Ndle - Smpl 18 mm 5/25 ml 16 ga (9.00 in)	209114	ea	*
Manifold - SS Solenoid Valve 4560	209494	ea	
Assy - Sparge Filter 4560	209254	ea	*
Battery - 3.6V Backup	211953	ea	*
Std - Detector / P&T 100 ppm / MEOH	218966	ea	*
Valve - Kel-F 3-Port Sample 10-32	218982	ea	
Valve - SS 6-Port Elect 1/16	219873	ea	
Kit - Startup 4560	220160	ea	
Thermocouple Assy - 7.25 in	227314	ea	
Needle Assy - Ndle Smpl (Side Hole 8.8 in)	227371	ea	*
Manual - Oper & Srvc 4560	227959	ea	
Wrench - Sparger 1-1/8 in OE	227975	ea	
Kit - Installation	228056	ea	
Transformer Assy - Trap 4560	228080	ea	
Frmwr (EPROM chips) - 4560	228106	ea	*
Internal Carrier Flow Option - 4560	227934	ea	
Gauge - Pressure 0-60 psi	202325	ea	
Tube Assy - Carrier FC / Gauge	227538	ea	
Tube Assy - Regulator In / Carrier FC In	227587	ea	
Flow Controller - SS 15 ml 1/16 RF	228031	ea	

Part Name	Part #	U/M	XPND
Sample Heater Option - 4560	225649	ea	
Lamp - Sample Htr 4560	225656	ea	*
Cable - I/O Board to Lamp	227082	ea	
Cable - I/O Board to Relay	227090	ea	
Assy - Sparge Door	230052	ea	
Assy - Lamp Cover	230060	ea	
Thermocouple - Needle Sparger Htr	272120	ea	