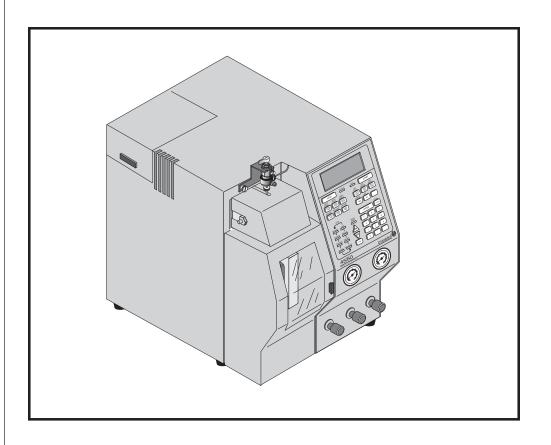


## Model 4560 Sample Concentrator Operator's Manual









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Revision 2.3—August 2002

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OI Analytical Part #227959



## **Limited Warranty**

OI Analytical warrants each Model 4560 Sample Concentrator against defects in materials and workmanship under normal use and service for a period of one (1) year. Equipment installed by OI Analytical is warranted from the installation date; all other equipment is warranted from the ship date. If purchaser schedules or delays installation more than 90 days after delivery, then warranty period starts on the 91st day from date of shipment. This warranty extends only to the original purchaser. OI Analytical will, at its option, repair or replace equipment that proves to be defective during the warranty period, provided the equipment is returned to OI Analytical at the expense of the purchaser.

Consumables, columns, lamps, and high temperature furnaces are warranted for 30 days (parts only) and are not available for coverage under extended warranties or service contracts.

This warranty shall not apply to defects originating from:

- Improper maintenance or operation by purchaser.
- Purchaser-supplied accessories or consumable.
- Modification or misuse by purchaser.
- Operation outside of the environmental and electrical products specifications.
- Improper or inadequate site preparation.
- Purchaser-induced contamination or leaks.

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

Any service requests or questions should be directed to the Technical Support Department at (800) 336-1911 or (979) 690-1711.



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## Chapter 1 Introduction

The Model 4560 Purge-and-Trap Sample Concentrator collects and transfers volatile organic compounds to a GC or GC/MS for analysis. It is specifically designed to purge volatiles from water, soil, and air for concentration onto a sorbent trap in compliance with USEPA protocol. The sample transfer line connects directly to the column or through an optional Low-Dead-Volume Injector<sup>TM</sup> to the GC column in an analytical system.

## **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 multisampler) 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" underreversed 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 analytes.

## **Features**

- Optional Infra-Sparge<sup>TM</sup> Sample Heater provides temperature feedback from within sample and safety shut-off.
- Built-in Cyclone Water Management<sup>TM</sup> minimizes the amount of water transferred to the analytical column and detector.
- Uses 5- and 25-mL frit and needle spargers and disposable tube spargers, using the same length needle and "wide-neck" 18-mm mount.
- Handles sample foaming and spillover through a removable particle foam filter in the sample inlet.
- Provides controlled temperature of each heated zone.
- Processor-controlled and fully interactive via the RS-232-C communication port.
- Basic operational sequence is controlled by key entry; operator interface is simple to use and vacuum-fluorescent display is easy to read.



- 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 reduces run times.
- All lines contacting the sample are inert and impermeable to gases.
- Incorporates power-up self-test and diagnostics messages.
- Optional Internal Carrier Flow Controller allows all gases (purge and carrier) to be provided by and monitored through the sample concentrator.
- Incorporates BCD inputs for multisampler position monitoring.

## **Specifications** 1

## **General Specifications**

## **Safety Certifications**

- UL-1262 Listing
- IEC-1010-1 classification by UL
- CSA 22.2 (classification pending)

#### **Dimensions**

- 14.5" H x 10.25" W x 14.2" D (37.12 cm H x 26.24 cm W x 36.35 cm D)
- Footprint 145 in<sup>2</sup>

#### Weight

• 31 lbs (14.06 kg)

## **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
- Trap oven size (to be cooled) < 25 in<sup>3</sup>
- Trap backpressure internally optimized
- Minimum cooldown: ambient +1°C

#### Valve

- Electrically DC 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 approx. 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
- All heated zones tested during self-test period

#### **Transfer Line**

- Fused-silica-lined stainless steel standard (all sample pathways)
- 48" standard
- 60" optional

## **Column Compatibility**

- 0.20–0.53 mm with Cryo-Focusing Module<sup>TM</sup> ( CFM<sup>TM</sup>)
- 0.32–0.53 mm (or packed) without CFM

#### Glassware

#### Standard

• 5-mL frit sparger (18-mm neck)

#### **Optional**

- 5-mL needle sparger (18-mm neck)
- 10-mL disposable test tubes (18-mm neck)
- 25-mL needle sparger (18-mm neck)
- 25-mL frit sparger (18-mm neck)

## **Performance Specifications**

#### **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

## **Trap**

- >900°C/min heating rate (25°C to 180°C in approximately 10 sec)
- >250°C/min cooling rate (180°C to 25°C in approximately 35 sec)
- Minimum cooldown: ambient +1°C

#### **Optional Infra-Sparge Sample Heater**

- Up to 30°C/min sample heating rate
- $\pm 1$ °C temperature accuracy for 5 or 25 mL samples



### **Temperature Accuracy**

•  $\pm 1^{\circ}$ C for *all* heated zones

## **Temperature Stability**

•  $\pm 1^{\circ}$ C for *all* heated zones

## Water Management

- Eliminates all but approximately 0.063 µL of trapped water/min of desorb\*
- Operates at *ambient* temperature
- Water removal at level equivalent to condensation at 4.8°C
- Polar compounds unaffected

#### **Communications**

## **Output Signals**

 2 sec contact closure at PURGE READY or PURGE END, 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-232-C (bi-directional)
- Baud Rate 9600
- Optional software package (WinTrap *Plus*)
- O•I•NET<sup>TM</sup> network interface for inter-instrument communication

## Requirements

## **Gas Requirements**

• 99.999% He or N<sub>2</sub> purge gas

## **Power Requirements**

- Standard unit: 110 VAC (+10%/-15%)/50/60 Hz (800 VA max)
- Standard unit operates at 100 VAC (+20/-5%)/50/60 Hz, for Japan
- Available unit for: 220 VAC  $(\pm 10\%)/50/60$  Hz)

#### **Fuse Requirements**

6.3 A 250 V fast blo for 110 and 220 VAC units

## **Major Options**

- Internal Carrier Flow Control (Part #227934)
- WinTrap Plus Softare
- Infra-Sparge Sample Heater (Part #225649)
- Cryo-Focusing Module (Part #210120)

<sup>\*</sup>Under normal USEPA Method 502.2 times and temperatures.

## **Safety Information**



The Model 4560 Sample Concentrator has been designed and tested in accordance with recognized safety standards and designed for use indoors. Using the instrument in a manner not specified by the manufacturer may impair the instrument's safety protection. Whenever the safety protection of the Model 4560 has been compromised, disconnect the instrument from all power sources and secure the instrument against unintended operation.

## **Operator Precautions**

For operator safety, pay attention to *WARNING* and *CAUTION* statements throughout the manual.

- A **WARNING** indicates a condition or possible situation that could result in physical injury to the operator.
- A *CAUTION* indicates a condition or possible situation that could damage or destroy the product or the operator's work.

Warnings and precautions in this manual or on the instrument must be followed during operation, service, and repair of the instrument. Failure to follow these warnings and precautions violates the safety design standards and intended use of the instrument. OI Analytical will not be liable for the operator's failure to comply to these warnings and precautions.

The Model 4560 must be connected to a dedicated AC power supply through a three-conductor power cord with the third wire firmly connected to an electrical ground at the power outlet. Any interruption of the grounding conductor or disconnection of the protective earth terminal could cause a shock that could result in personal injury.

## **General Precautions**

- Disconnect the AC power cord before removing covers (excluding the carousel cover).
- Replace or repair faulty or frayed insulation on power cords.
- Perform periodic leak checks on supply lines, fittings, and pneumatic plumbing.
- Arrange gas lines so they can not become kinked, punctured, or otherwise damaged, and will not interfere with foot traffic.
- Turn off the main power switch and disconnect the main power cord before using a liquid solution to locate leaks.
- Wear safety glasses to prevent possible eye injury.



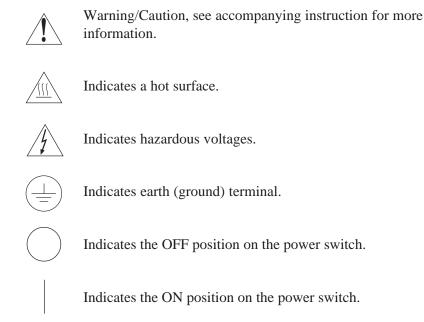
Do not perform unauthorized modifications or substitute parts that are not OI
 Analytical original parts to the instrument. Any unauthorized modifications or
 substitutions will void the warranty.

## **Compressed Gas Cylinder Precautions**

- Compressed gases should be stored and handled strictly in accordance with relevant safety codes.
- Fasten all cylinders securely to an immovable structure or permanent wall.
- Store or move cylinders only in a vertical position. Do not move or transport cylinders with the regulators attached.
- Use only approved regulators and tubing connections.
- Connect cylinders to instruments with pressure ratings that are significantly greater than the highest outlet pressure from the regulator.
- Nitrogen and helium have been identified as asphyxiants. These gases and the
  cylinders containing them should be handled and stored in a manner consistent
  with OSHA regulations. Adequate ventilation should be maintained in areas
  where these materials are used and store. The analyst should avoid prolonged
  exposure to high concentrations of these gases.

## **Safety Symbols**

The following symbols may be located on the instrument:





# **Chapter 2 Description of Components**

## **Exterior Description of Components**

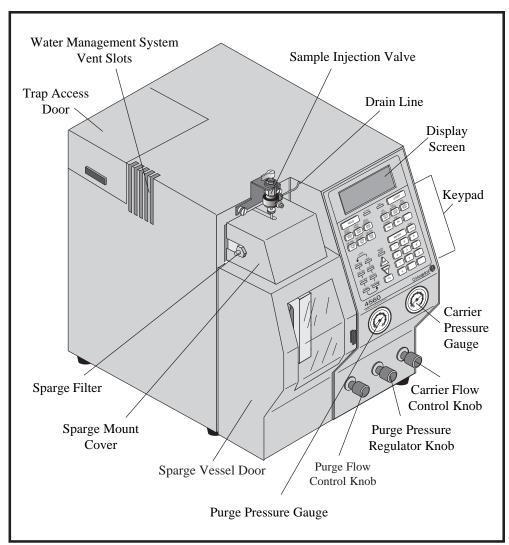


Figure 2.1. Model 4560 Sample Concentrator

**Carrier Flow Control Knob** (optional) adjusts column carrier flow from the Model 4560. The controller is supplied with a 0–15 mL/min restrictor (for most capillary column applications).

**Carrier Pressure Gauge** (optional) displays carrier (head) pressure from 0–60 psi (0–400 kPa). This gauge, along with the corresponding carrier flow controller, is optional. The option allows easy routing of all necessary gases (carrier and purge) to the Model 4560, eliminating the need for GC pneumatics in cases when access to the GC pnuematic bay is limited (e.g., GC/MS).



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

**Drain Line** drains a water sample automatically during DESORB. This flexible line runs through a pinch drain valve in the interior right bay of the Model 4560, which opens to drain the sample. The drain line leads to the sample drain port on the back panel of the Model 4560 and is connected to the sample injection valve by a knurled <sup>10</sup>/<sub>32</sub>" nut and stainless steel ferrule.

**Keypad** (tactile elastomeric) is used to enter settings and select parameters displayed on the display screen.

**Purge Flow Control Knob** adjusts the flow of gas supplied by the purge gas pressure regulator. Turning this knob counterclockwise increases the amount of purge gas passed through the sample each minute (usually 30–40 mL/min).

**Purge Pressure Gauge** displays the 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 (see "Model 4560 Diagnostics Tools" in Chapter 5 for expected pressures).

**Purge Pressure Regulator Knob** adjusts incoming purge gas pressure supplied 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.

**Sample Injection Valve** is used to manually syringe-inject (into a Luer-Lok® injection port fitting) a water sample into the sparge vessel. This valve is also used to connect the drain line if frit is sparging or the purge gas line if the needle is sparging.

**Sparge Filter** protects the Model 4560 trap and interior components from foaming samples, which are often encountered in purge-and-trap analysis. It can be replaced as an expendable part or cleaned via the procedure outlined in this manual. If an OI Analytical Discrete Purging Multisampler (DPM-16) is present, the sparge filter is removed and the port is used to connect the heated transfer line from the multisampler.

**Sparge Mount Cover** provides a mount for the sparge vessel and insulation for the sample mount heater. This cover slides forward to expose the sample inlet and forward valve oven area.

**Sparge Vessel Door** (optional)is provided with the Infra-Sparge Sample Heater Option for safety and to aid in sample heating. The operator can visually monitor the sample through the door's window.

**Trap Access Door** allows access to the Model 4560's trap without removing the instrument cover. When this door is opened, a safety shutoff switch cuts power to the trap.

Water Management System Vent Slots aid in cooling the water management zone before desorption begins. These slots should not be covered during operation.

## Model 4560 Back Panel

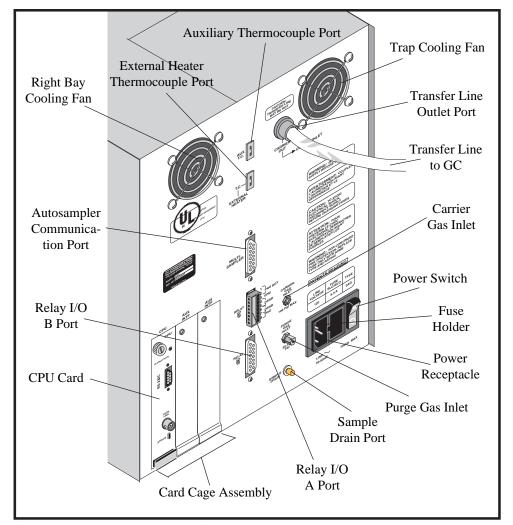


Figure 2.2. Model 4560 Back Panel

**Autosampler Communication Port** (15-pin D-sub connector) provides communication line connection from the Model 4560 to any one of several OI Analytical autosampler products (see "Communication Cabling" in Chapter 3).

**Auxiliary Thermocouple Port** (Accepts Type K thermocouple) monitors the temperature of a selected heated zone. It occasionally monitors the oven temperature to signal a "GC Ready" to the Model 4560 when no "GC Ready" contact closure is available from the GC.

**Card Cage Assembly** contains slots for the insertion or removal of the CPU card and two auxiliary interface cards. The CPU card allows for connection of an RS-232-C cable, has a port used for data linking, and contains status LEDs used for CPU card troubleshooting.

**Carrier Gas Inlet** connects the ½16" carrier gas supply line from an external flow controller (e.g., the GC injector flow controller) to the Model 4560. Supply carrier gas at 100 psi **maximum** pressure. This inlet remains plugged when the Model 4560 Internal Carrier Flow Control Option is present.



**CPU Card** is located in the first slot of the card cage assembly. It controls all Model 4560 functions and measures and controls all Model 4560 heated zone temperatures. This card also communicates with the operator by controlling the front panel keypad and display. The RS-232-C port is accessed through the 9-pin connector on the back of the CPU card. This card contains the Model 4560 EPROM chips.

**External Heater Thermocouple Port** (Type K thermocouple) monitors the temperature of an external heated zone.

**Fuse Holder** contains the main fuse that protects the Model 4560 from a short circuit condition.

**Power Receptacle** connects the Model 4560 to an appropriate power source via a cable provided in the start-up kit.

**Power Switch** is the power control switch. Turning the unit on is accompanied by a power-up self-test.

**Purge Gas Inlet** connects the ½" purge gas line from the secondary regulator at 30–100 psi. (The purge gas inlet also supplies carrier gas to the GC when the Internal Carrier Flow Control Option is present on the Model 4560.)

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

**Relay I/O A Port** (8-pin Phoenix connector) provides hard-wire communication interface from the Model 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.

**Relay I/O B Port** (15-pin D-sub connector) provides communication interface from the Model 4560 to any GC system providing and accepting the required contact closures. The standard interface cable provided connects to another interface cable for a specific GC brand (ordered separately).

**Sample Drain Port** is a fitting that connects to the drain line provided in the start-up kit. Route the drain line to an appropriate-sized waste receptacle.

**Transfer Line to Gas Chromatograph** is a heated line used to carry volatiles desorbed from the trap to the GC. Temperature is operator-selectable.

**Transfer Line Outlet Port** allows the transfer line to exit the Model 4560 and provides strain relief.

**Trap Cooling Fan** cools the Model 4560's trap, as required, during the purge-and-trap cycle.

## **Infra-Sparge Sample Heater Option**



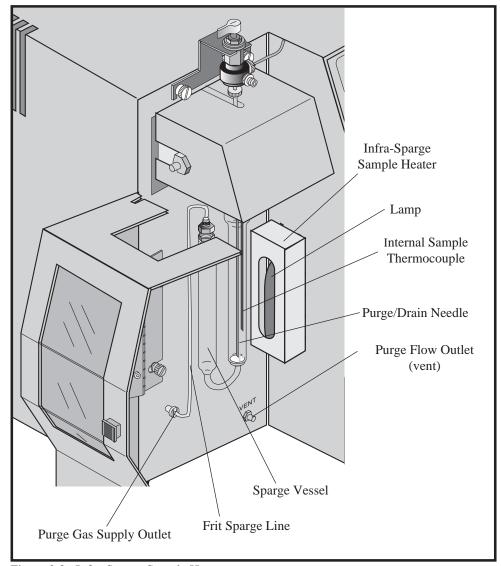


Figure 2.3. Infra-Sparge Sample Heater

**Frit Sparge Line** connects to the purge gas supply fitting and runs to the purge inlet arm of a frit-style sparger. This line supplies purge gas to the frit when frit sparging.

**Infra-Sparge Sample Heater** is a high-intensity bulb that heats water or soil samples. It has a removable aluminum cover with built-in filter to partially block visible light. It is controlled and monitored through temperature feedback from inside the sample.

**Internal Sample Thermocouple** provides direct sample temperature feedback to the sample heater for temperature monitoring and control of water or soil samples.

**Lamp** is an expendable component replaced when necessary only by OI Analytical trained technical support.



**Needle Sparge Line** (not shown) connects to the purge gas supply outlet and runs to the sample injection valve when needle sparging. This line supplies purge gas to a needle-style sparger through the sample purge needle.

**Purge/Drain Needle** supplies purge gas directly into the sample when needle sparging. It acts as a drain pathway when frit sparging. If removed, the sparge mount must be appropriately plugged to close the system (e.g., when performing air-tube desorption with the Model 4560).

**Purge Flow Outlet** is the purge gas flow vent. The purge flow can be adjusted or monitored without removing the Model 4560 cover. A purge vent plug (Part #248864) is provided to plug this outlet when performing a purge gas leak check.

**Purge Gas Supply Outlet** connects the frit-style or needle-style purge gas line to the appropriate sparge vessel.

**Sparge Vessel** is a reservoir for sample purging. Vessels are available in a 5-mL, 10-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).

## **Interior Components** I

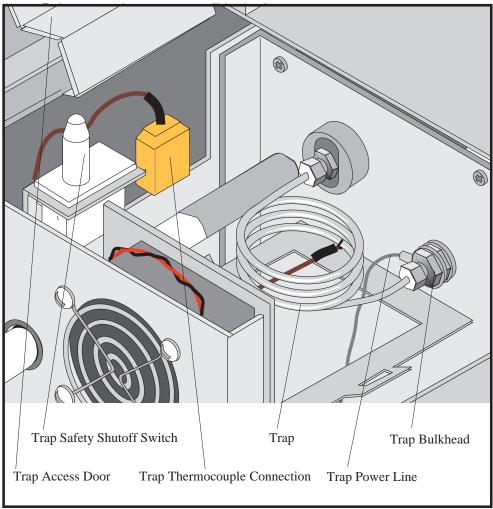


Figure 2.4. Interior Components of the 4560 Trap Oven

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

**Trap Access Door (partially shown)** is a metal cover that must be in place over the trap to ensure proper trap cooling. If the trap cooling rate is insufficient, a warning message will be displayed.

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

**Trap Power Line** provides current directly to the trap bulkhead to heat the trap through direct-resistive heating.

**Trap Safety Shutoff Switch** interrupts power to and cools the trap when the trap access door is opened.

**Trap Thermocouple Connection** provides trap temperature feedback to the I/O board on the Model 4560.



CAUTION:

The trap may remain hot for several minutes after opening the trap access door if the trap was at an elevated temperature.

## Notes





# **Chapter 3 Installation**

This chapter describes the basic installation procedure of the Model 4560 Sample Concentrator.

Failure to complete all sections of this installation procedure may result in incomplete installation and improper operation of the Model 4560.

## Materials Needed

The following items required for installation are not provided with the Model 4560 start-up kit (Part #220160).

- Purge Gas Source: A source of purge gas (99.999% pure He or N<sub>2</sub>), regulated to a pressure between 30 and 100 psi (207–1724 kPa), connects to the purge gas inlet (50 psi is recommended). External gas scrubbing is highly recommended. Plumb solvent-rinsed, baked copper tubing to within 5 ft of the Model 4560 (see Gas Hookup Lines).
- **Gas Hookup Lines:** A clean stainless steel or copper ½" (3.2 mm) O.D. gas hookup line connects the purge gas source to the purge gas inlet of the Model 4560 (5 ft of precleaned copper tubing is provided).
- Carrier Gas Source: A source of carrier gas must be provided to the flow controller of the GC interfaced to the Model 4560. 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 Model 4560, the purity requirement of this gas is the same as for the purge gas, and the allowable pressure range is 0–100 psi (0–1724 kPa). This carrier gas may be from the same source as the purge gas. When the Internal Carrier Flow Option is present, it is only necessary to connect a single gas source (purge) to the Model 4560.
- GC Interface Kit: A kit for interfacing the Model 4560 to the GC is needed for connecting the GC carrier gas outlet to the Model 4560 carrier gas inlet and for connecting the Model 4560 transfer line to the GC injection port.
- **Power Source:** A standard receptacle for 110 VAC (+10/-15%) power capable of providing 8 amps or 220 VAC (±10%) at 4 amps is required. The power cord provided is 6 ft (1.83 m) long.



## **Unpacking and Positioning the Instrument**

Unpack the instrument(s) and check the items against the component list. If any damage is apparent, notify the carrier immediately. Save all packing materials until proper operation of all components is verified.

**Note:** All instruments that are returned to OI Analytical for service or warranty repair must be shipped in the instrument's original OI Analytical box with its packing materials. *If instruments are damaged due to improper shipping, OI Analytical will not be responsible for the cost of repairs.* If there is no access to proper shipping materials, contact the OI Analytical Order Entry Department at (800) 336-1911 or (979) 690-1711.

- 1. Remove the Model 4560 from the shipping carton.
- 2. Position the Model 4560 on the side of the GC nearest the GC injection port. The space under the instrument should be clear of obstructions for proper air flow during operation. Placing the Model 4560 on a hot surface may inhibit proper trap cooling and affect results. The standard 48" sample transfer line may be exchanged for an optional 60" line if longer transfer to the GC is necessary.

## **Sparge Vessel (Sparger) Installation**

The Model 4560 arrives configured with a 5-mL frit sparger (see Figure 3.1). For needle sparging, the Needle Sparge Hardware Kit (Part #227397) is required. To convert back to a frit sparger, see "Cleaning/Changing the Sparger" in Chapter 5.

## **Needle Sparger Installation**

A Needle Sparge Hardware Kit (Part #227397) is available as a Model 4560 option. A needle sparger is designed for 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 USEPA methods using the Infra-Sparge Sample Heater option.

- 1. Remove the frit sparger from the Model 4560.
- 2. Disconnect the frit-style purge gas line (Part #227652) from the front panel of the Model 4560 and replace it with the needle-style purge gas line (Part #227389) provided. Leak-check the fitting.
- 3. Route the purge gas line through the slot provided in the back left cover of the sparge door (if present) to the black sample injection valve normally used for direct injection.
- 4. Disconnect the drain line from the sample injection valve; it is not needed for needle sparging (the line may loosely rest unconnected).



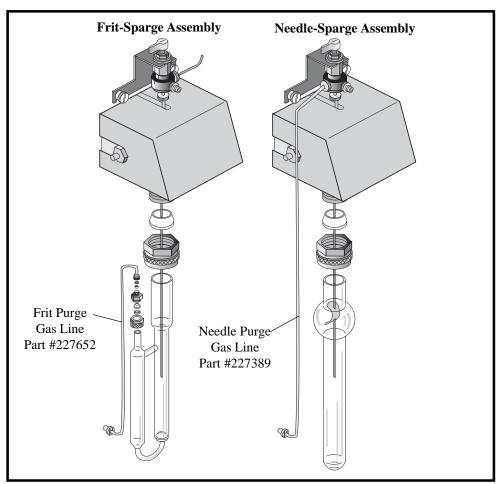


Figure 3.1. Sparge Vessel Assemblies

- 5. Loosen the sample injection valve from the bracket, rotate the valve  $90^{\circ}$  clockwise, and retighten.
- 6. Remove the Luer-Lok injection adapter from its old position and thread the Luer fitting into the frontmost port on the sample injection valve.
- 7. Connect the needle-style purge gas line to the 9 o'clock position on the sample injection valve and finger-tighten.
- 8. Slide a Mud-Dawg $^{\text{TM}}$  over the purge needle following instructions provided with the Mud-Dawg.
- 9. Slide the appropriate needle sparger over the purge needle into the 18-mm knurled nut and finger-tighten. Leak check the connection.



## **Gas Connections**

- 1. Connect the ½" purge gas line between the purge gas source and the receptacle marked "Purge Gas Inlet" on the back of the Model 4560. Use the standard ½" stainless steel or brass Swagelok® nut and ferrule provided. If the Internal Carrier Flow Control option (Part #227934) is present on the Model 4560 (i.e., if a carrier gas flow control knob and gauge is present on the Model 4560), this connection is all that is necessary to provide both purge and carrier gas to the analytical system. If the option is present, **do not** remove the plug from the carrier gas inlet on the back of the Model 4560.
- 2. If the Internal Carrier Flow option is not present, connect the carrier gas interface line (Part #188466-002) between the GC flow controller and the carrier inlet receptacle on the back of the Model 4560. The ½16" nut and ferrule attached to the line connect to the Model 4560.

## **Communication Cabling**

The following figures depict communication cabling for several standard instrument configurations. All figures show a back view.

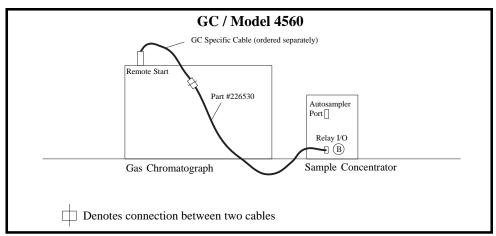


Figure 3.2. Communication Cabling Between a GC and the Model 4560

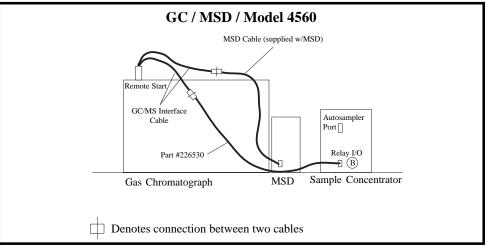


Figure 3.3. Communication Cabling Between the GC/MSD (DOS/UNIX)/Model 4560



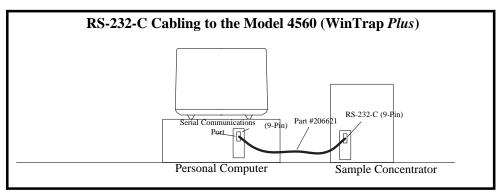


Figure 3.4. Connection of Serial Communications Port on PC to the Model 4560

## **Electrical Connections**

- 1. Verify the voltage requirement (110 VAC  $\pm$ 10/-15% or 220 VAC  $\pm$ 10%) for the unit to be installed. Plug one end of the power cable (Part #116038) into the Model 4560 power receptacle and the other end into an appropriate power outlet.
- 2. Provide power to any OI Analytical autosampler to be connected to the Model 4560 by following the instructions in the appropriate autosampler Operator's Manual.

## **Transfer and Drain Line Connections**

## **Connecting the Transfer Line to the GC Injector**

The heated transfer line on the Model 4560 connects directly to the analytical column via a union connecting the ½16" transfer line to the appropriate column size. OI Analytical manufactures a Low-Dead-Volume (LDV) Interface (for existing GC injectors) or a Low-Dead-Volume (purge-and-trap) Injector (LDVI) to make this connection and still retain direct injection capability onto the column. For either of these options, refer to the appropriate Installation and Maintenance Guide for installation instructions; otherwise, interface the transfer line to the column, minimizing dead volume.

## **Connecting the Autosampler Transfer Line to the Model 4560**

The autosampler transfer line interfaces easily to the sample inlet mount of the Model 4560. To connect the transfer line, follow these steps (see Figure 3.5):

- 1. Remove the sparge filter by turning it counterclockwise with a 3/8" wrench.
- 2. Replace the sparge filter with the A/S interface assembly (Part #209536) provided in the autoampler's start-up kit.



3. Connect the transfer line to the A/S interface adapter using the ½16" Valco® male nut and ferrule provided. Wrenchtighten and leak check at the sample inlet mount.

Note: Connecting the autosampler transfer line prevents the use of the single sparger position on the Model 4560.

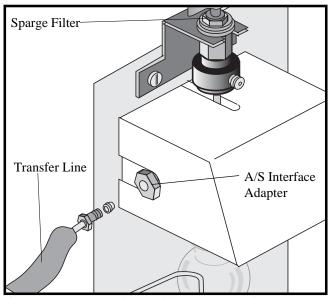


Figure 3.5. Autosampler/Model 4560 Connection

## **Drain Line Connection**

To connect the sample drain line (Part #166224) supplied in the start-up kit, press one end of the line onto the barbed Luer-lock fitting marked "Sample Drain" on the back panel of the Model 4560. Route the other end to an appropriate drain or receptacle.

After completing installation, go to "Pre-Run 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 Model 4560 Sample Concentrator.

The Model 4560 strips purgeable organic compounds from an aqueous sample, concentrates them on a specified sequence of adsorbent materials, and rapidly desorbs them using heat and carrier gas flow onto a gas chromatographic column. The Model 4560 is one component of an analytical system for purge-and-trap analysis. Other necessary components include a gas chromatograph (GC), single or multiple detectors, and a data-handling device for quantifying detector signals. An autosampler, for introducing samples to the concentrator, can also be part of the overall analytical system.

## **Instrument States**

There are 11 primary states. A state is a set of valve and trap temperature combinations defined for 4560 analyses:

STANDBY PURGE COMPLETE
PURGE READY DESORB READY
PREPURGE DESORB PREHEAT

PREHEAT DESORB (W/ or W/O DRAIN)

PURGE (ADSORB) BACKFLUSH BAKE (W/ or W/O PURGE)

**DRY PURGE** 

The Model 4560 microprocessor runs through specific sequences of these states. Various analytical procedures for purgeable organics require different times and temperatures for these states, so the Model 4560 can be programmed for up to 14 operator-defined parameters, or methods.

Each unit is shipped from the factory set to Method 1, which corresponds to parameters included in USEPA Method 502.2/524.2.

## **State Sequencing** 1

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



STANDBY - The Model 4560 remains in STANDBY until attaining all temperature set points. When all heated zones are in a ready state and the trap has cooled to the purge temperature set point, the Model 4560 advances to PURGE READY. STANDBY is one of several states not indicated by an LED on the front panel of the Model 4560, and is only seen on the display screen since no parameter entry is possible.

PURGE READY - This is a pass-through state indicating that all conditions are met to start the next analysis. When manually syringe-injecting into the Model 4560 (no autosampler 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.

**PREPURGE** - This is an optional state normally used only for air analysis. Prepurging an air tube removes oxygen and excess moisture accumulated on the tube during sample collection. Prepurging a hydrophobic substrate (e.g., Tenax<sup>®</sup>) reduces the moisture transferred to the trap.

**PREHEAT** - This is a settable time prior to purge allowing a heated sample (e.g., air tube, soil, or water) to equilibrate to a preset temperature before purge begins. This state is only used when a sample heater is present and sample heating is desired.

**PURGE (ADSORB)** - This is the sample extraction state. Inert gas passes through the sample for a specified time and temperature, and volatiles are adsorbed onto the Model 4560's trap for subsequent desorption to the GC column.

**DRY PURGE** - This is an optional state that removes moisture from the hydrophobic substrates in the trap. In DRY PURGE, the sample is bypassed and the trap is purged with dry purge gas directly. DRY PURGE is usually not necessary with an effective water management system.

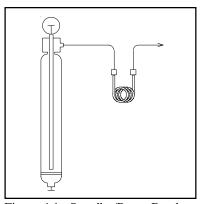


Figure 4.1. Standby/Purge Ready

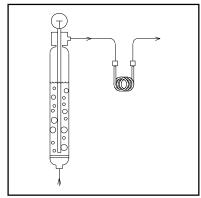


Figure 4.2. Purge (Adsorb)

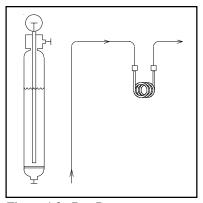


Figure 4.3. Dry Purge

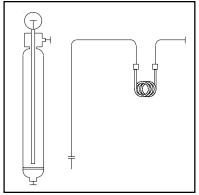


Figure 4.4. Purge Complete/ Desorb Ready

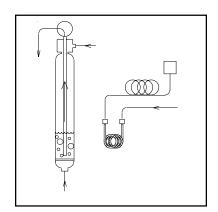


PURGE COMPLETE - This is a pass-through state indicating that the purge state is complete. If the water management system is "NOT READY," the Model 4560 remains in PURGE COMPLETE until the Cyclone Water Management System reaches the required set point for desorption.

**DESORB READY** - After completing all purging (PURGE and DRY PURGE), the Model 4560 advances to DESORB READY. DESORB READY is a diagnostic state where the Model 4560 checks for ready signals from other components of the system (e.g., GC) before advancing to DESORB.

#### **DESORB PREHEAT** - If DESORB PRE-

HEAT is enabled, the Model 4560 heats the trap to a desorb preheat set point under no carrier gas flow (e.g., prior to valve rotation). This technique is sometimes used to narrow desorption bandwidth when trap heating (analyte release) from the trap is slow. Desorb preheat is usually not necessary due to the Model 4560's direct-resistance heating.



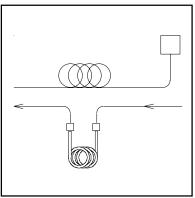


Figure 4.6. Backflush Bake

**DESORB** - During DESORB, the Model 4560's trap heats rapidly to the set temperature, transferring volatile compounds through the Model 4560 heated transfer line to the GC injector port. If a discrete multisampler is used (draining is not possible) and the Model 4560 is configured not to drain, no sample drain will be attempted (DESORB WITHOUT DRAIN). Otherwise, an aqueous sample automatically drains during the DESORB state (DESORB WITH DRAIN).

**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. Choose whether to purge or not purge the sample during BAKE (see "Configuration Key" in this chapter).

**Note:** Other autosampler-specific states may be present. Refer to the appropriate autosampler operator's manual for descriptions of any states not listed here.



## **Cycle State Indicator**

The Cycle State Indicator on the Model 4560 front panel displays instrument activity. Following are guidelines for reading the Cycle State Indicator.

- Illuminated **orange** lights indicate which states have been activated during a run.
- Illuminated **green** lights indicate the current state.
- A STANDBY state, not shown on the cycle state indicator, occurs between BAKE and PURGE READY. During STANDBY the trap temperature equilibrates to the PURGE temperature set point.

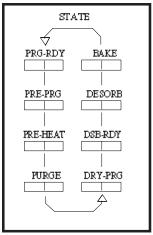


Figure 4.7. Cycle State Indicator

 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 may be cooling before DESORB during this state.

## The Model 4560 Keypad

The Model 4560 keypad has eight key types, which are explained in detail in "Model 4560 Key Descriptions" later in this chapter. To access parameters labeled on the keys, simply press the appropriate key. To access 2nd functions, written in maroon above the keys, press the [2nd] function key (the maroon key) and then the appropriate selection. See Figure 4.8.

The next section explains how to run a basic sample on the Model 4560; for custom configuration, see the appropriate instructions under "Method Files"; for detailed key information, see "Model 4560 Key Descriptions."

## **A Brief Overview**

The following instructions provide a fundamental overview of the procedure for Model 4560 daily analyses. The addition of autosamplers and options will add a step or two. Detailed step-by-step instructions follow in the next section.

- 1. Adjust gas pressures, if necessary.
- 2. Turn on the Model 4560, if necessary.
- 3. Wait for the self-test to run. If the self-test fails, identify and solve the problem. If the self-test passes, continue.
- 4. Perform all necessary functional checks.



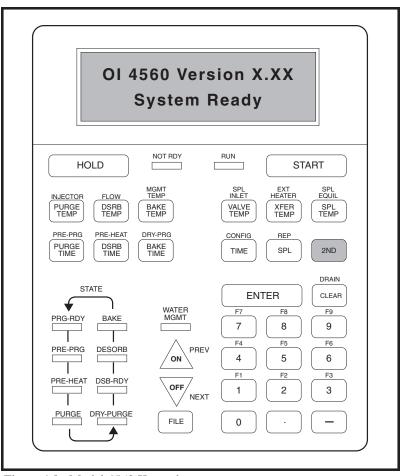


Figure 4.8. Model 4560 Keypad

- 5. Choose a presaved method file (e.g., Method 1, USEPA 502.2/524.2) *or* create a new method file by resetting times/temperatures according to instructions.
- 6. Load the sample.
- 7. Press [START] to begin the run.



## **Pre-Run Functional Checks**

Perform the following steps to confirm proper function of the Model 4560 before sample analysis. Refer to Chapter 6, "Troubleshooting," to use these checks to isolate problems.

## **Pre-Power-Up Checks**

- 1. Adjust purge and carrier gas pressures (purge pressure should be set to 20 psi in STANDBY or PURGE READY).
- 2. Confirm that carrier gas flow is set to the desired rate.
- 3. Turn on the Model 4560 power switch. The Model 4560 will perform a self-diagnostic test for 60 seconds.

## The Model 4560 Self-Test

The Model 4560 automatically performs a self-diagnostic test upon power-up. The self-test temporarily changes the set point for the heated zones to 25°C above ambient temperature. At the end of the self-test, all zone readings will be compared to the set point (ambient +25°C) and the error for each zone will be computed internally. Heated zones tested are as follows:

- Trap
- External Heater
- Sample Inlet
- Control Manifold

- Valve
- Transfer Line
- Sample Heater

Any zone not within its valid range at the end of the self-test causes 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 depend 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:

- A safety switch will not allow a zone to heat (e.g., trap and/or sample heater).
- An open thermocouple connection has caused an over-temperature condition.
  If the over temperature LED on the I/O board is "ON," power is shut off to all heated zones.
- The fuse for the heated zone is blown.



• The thermocouple is shorted for the failed zone. The zone will heat, but readings never change.

If the unit fails the self-test, you will be prompted to press [2nd] [9] to display the errors or failure conditions found. Then, a prompt to press [ON] or [OFF] will display the error if only one error occurred, or will scroll through multiple error messages.

Bypass the self-test at any time by pressing any key on the Model 4560 keypad during the test. At the end of the self-test, the unit restores all settings to their previous values.

## **Confirming 6-port Trap Valve Rotation**

- 1. Press [2nd] [1] to show the 6-port valve position.
- 2. Test 6-port valve rotation by toggling the trap to the GC [ON] and [OFF]. Trap to GC ON = Desorb. Trap to GC OFF = Purge.
- 3. Verify that you hear the valve actuate.

## **Setting the 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:

- 1. While the system is in STANDBY or PURGE READY, set the purge gas pressure (using the knob on the Model 4560 front panel) to read 20 psi, as indicated on the purge pressure gauge also on the front panel.
- 2. Confirm that the Model 4560 is not in a RUN state (e.g., the green "RUN" LED is off). If the "RUN" LED is on, press [HOLD] to exit the RUN state.
- 3. Press [OFF] to advance to the next state (PURGE).
- 4. Measure the purge flow from the purge vent under the Model 4560 front panel sparger, using a flowmeter accurate to within 1 mL/min.
- 5. Adjust to the desired flow rate using the purge flow controller on the Model 4560 front panel.
- 6. Confirm that the purge pressure in PURGE is 6–11 psi.

**Note:** Purge flow rate can have a significant effect on the recovery and extraction efficiency of some analytes. Experimenting with purge flow rate is an important factor in optimizing analyses.

7. Return to STANDBY by pressing [2nd] [ON] [ENTER], and confirm the return to system pressure (20 psi).



## **Purge Gas Leak Check**

Confirm that the purge gas flow system is leak-free:

- 1. While the system is in the PURGE state, inject an appropriate-sized sample of clean water, using the sample syringe (see "Syringe Sample Loading" in this chapter for loading instructions), and confirm visible purging.
- 2. Stop the purge flow from the bake valve by plugging the vent line end with the purge vent plug assembly (Part #248864) provided in the Model 4560 start-up kit.
- 3. Confirm that the purge gas pressure rises to 20 psi and that visible purging eventually stops. The rate will decay after several minutes. If 20 psi is not achieved within several minutes, check seals with an electronic gas-leak detector to find possible leaks.
- 4. With the system pressurized at 20 psi (e.g., 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 one minute period.
- 5. Remove the purge vent plug to regain purge flow.
- 6. Return to STANDBY by pressing [2nd] [ON] [ENTER].
- 7. Readjust purge pressure to 20 psi.
- 8. Press [OFF] to advance to PURGE.
- 9. Confirm the return of purge gas pressure to 6–11 psi.

### **Drain Check**

Check for proper sample drainage from the sparge vessel:

- 1. Leave the sample in the sparge vessel, and confirm purge flow rate and leak-tightness (see "Purge Gas Leak Check").
- Confirm the proper sample valve position for drain (see Figure 4.9). Press [2nd] [CLEAR] to drain. Verify drain by pressing [ENTER].
- 3. Measure the time it takes to drain the 5-mL sample (about 8–10 seconds is normal). If drain time is significantly slower than this, inadequate gas flow to the sparge vessel, improper sample

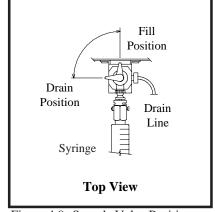


Figure 4.9. Sample Valve Positions

valve handle position, or a restriction in the drain line is probable. Thirty seconds should be sufficient for draining a 25-mL sample.

## Carrier Gas Leak Check



- 1. Disconnect the transfer line from the GC injection port. Take necessary precautions to prevent column or detector damage from the absence of carrier flow.
- 2. Block the transfer line end with a septum to completely stop flow.
- 3. Observe that the GC flow controller pressure (through the GC gauge or front of the Model 4560) climbs rapidly to full scale or to regulator delivery pressure.
- 4. Correct any leak problems as necessary to achieve proper flow at the transfer line end.

## Trap Bake

Upon installing a new trap, or if the Model 4560 has been idle for a period of time without cycling, bake the trap before performing a sample analysis.

- 1. Press [2nd] [OFF] to advance to BAKE.
- 2. Watch the temperature display to confirm that the trap temperature rises rapidly to the bake temperature set point. Any sample in the sparge vessel will automatically drain, then the Model 4560 will cycle to BAKE.
- 3. The system bakes the trap until the programmed BAKE time is completed, or press [HOLD] during a run to stop the cycle clock for extended baking.
- 4. After trap BAKE is complete, advance the system to STANDBY by pressing [2nd] [ON]. Verify by pressing [ENTER]. The system will return to STANDBY automatically.

After completing all functional checks, choose the method file you will use.

## Running a Sample

The following are step-by-step procedures to program the Model 4560 for daily analyses

#### **Method Files**

Every Model 4560 is shipped from the factory set to Method 1, which corresponds to parameters included in **USEPA Method 502.2/524.2**. If these are the parameters you need, press the [File] key to access the File Menu.

The [FILE] Key accesses GET, SAVE, and DELETE (DEL) method files used in the Model 4560. Access GET, SAVE, and DEL by selecting the appropriate



number key. In this case, select GET, choose Method 1, then [ENTER]. Go to *Syringe Sample Loading* for further instructions, or continue for custom file creation.

FILE [1] = Get [2] = Save [3] = Del

#### **Custom File Creation**

If your analyses require different set points than those in Method 1, this section explains how to create custom files.

#### To create custom files for different analyses:

 Scroll through and change temperatures and times to the desired set points. For detailed key explanations, refer to *Model 4560 Key Descriptions* later in this chapter.

#### To save custom configurations:

- 1. After selecting the desired times and temperatures, press the [FILE] key and select [2] to access the SAVE FILE display.
- 2. The SAVE FILE display saves set points under a given name. Select a file number by scrolling with the [ON]/[OFF] keys, then name and save the file by one of the following three methods:
  - Use the number keys to name an undefined file or rename an existing file, then press the [ENTER] key to save the set points. When a file is named, but not saved within five seconds by pressing [ENTER], the Model 4560 defaults to the File Menu Display.
  - Press only the [ENTER] key 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."
  - Press only the [ENTER] key 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 set points under file "2: 601-602."

When using the [ENTER] key to save a file, the message "FILE SAVED" indicates new set points were saved under the selected file.

**Note:** Saving new set points over an existing file replaces all set points currently defined in the selected file.

## **Retrieving Method Files**



- 1. Press the [FILE] key and select [1] to access the GET FILE display.
- 2. Select a file using the [ON]/[OFF] keys followed by [ENTER] to load the file, or [CLEAR] to return to the file menu. One of the following three messages will display upon pressing [ENTER]:
  - FILE LOADED The selected file set points have been successfully loaded and are now current.
  - NO FILE HERE A file was selected that contained no set points (i.e., an undefined file).
  - SAVE CURRENT SETTINGS? Y/N Indicates current set points have been changed but not saved. Use the [ON]/[OFF] keys to select Y (yes) or N (no) followed by [ENTER]. Selecting N ignores the changed set points and loads the selected file. Selecting Y allows the changed set points to be saved under a new or existing file. The display changes to the SAVE FILE mode. Once the set points are saved, the selected file is automatically loaded.

## **Deleting Method Files**

- 1. Press the [FILE] key, then [3], to reach the DEL FILE menu.
- 2. Select the file you wish to delete using the [ON]/[OFF] keys, then [ENTER]. This erases the file name and all set points, making the file undefined.

**Note:** Method File 1, which contains the set points for EPA Method 502.2/524.2, is stored as a protected file. **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 (times and temperatures) without changing the system configuration.

#### **Linking Multiple Method Files for an Autosampler Run**

The Model 4560 can internally download up to five selected methods during any automated (multisampler) run. For example, if there are 15 samples to run in a sequence, but not all by the same sample concentrator method, you may select up to five different saved methods and specify the number of samples to be run under each method.

1. Press [F3] Sequencing [2nd] [3].

The unit will display the Main Sequencing Screen:

Sequencing OFF [1] [2] [3] [4] [5]



The OFF designation indicates that sequencing is currently inactive and that the Model 4560 will run the currently loaded method until a new method is *manually* downloaded. Numbers [1]–[5] represent method entries that compose the sequence.

2. When [1] is pressed, the unit will display:

#1 File Name 1: 502.2/524.2

- 3. Select the first method to run in the sequence by scrolling through the list using the [ON]/[OFF] keys and pressing [ENTER]. Press [CLEAR] to return to the above screen. Repeat these steps for methods [2]–[5], as necessary.
- 4. Once complete, press [CLEAR] until reaching the Main Sequencing Screen. Then press [ON] to activate sequencing. The number of methods entered will be verified on the display:

Sequencing On Ready #1-#5

If the displayed numbers do not match the actual number of methods to be sequenced, select an undefined file from the File menu for undesired sequence numbers. All sequence files initially default to 502.2/524.2.

5. Press [CLEAR] to exit sequencing then proceed with sample preparation.

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

During a run, if a sample in the sequence is aborted for any reason, the sequence may continue at the same sample position by pressing [START] again. To reset a sequence:

6. Return to the sequencing display by pressing [2nd] [3], press [OFF], then [ON], then exit sequencing (press [CLEAR]), and press [START] once again.

**Note:** When sequencing files, always verify that multisampler Start/End positions **saved in each file** do not overlap from file to file.

Complete the above functional checks and method sequencing before continuing. The GC column and detector must also be properly interfaced, conditioned, and ready for sample introduction. Continue with the following instructions to load a sample.

## **Syringe Sample Loading**



- 1. Allow the sample to reach ambient temperature before loading it into the syringe. Purging a sub-ambient sample will significantly affect purge (extraction) efficiency.
- 2. Remove the plunger from the sample syringe.
- 3. 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.
- 4. Replace the syringe plunger and compress the sample.
- 5. Vent any residual air in the syringe while adjusting to the appropriate sample volume.
- 6. Add any applicable surrogate spiking solution and internal standard spiking solution through the syringe exit port using a 10 μL syringe, if necessary.

### **Syringe Sample Injection (Manual Injection)**

- 1. When the system is in PURGE READY, press the [START] key to begin the analysis.
- 2. Attach the syringe to the Model 4560 sample injection valve syringe port (see Figure 4.10).
- 3. Turn the sample valve handle to the **fill** position (see Figure 4.9).
- 4. Inject the sample into the sparge vessel and return the valve handle to the **drain** position. Remove the syringe. The sample will begin purging for the time set by the purge time set point. The system will then cycle through the states according to the set point values.

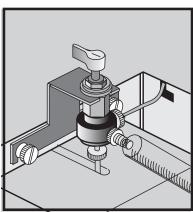


Figure 4.10. Syringe Injection

- 5. After the Model 4560 cycles to BAKE, rinse the sparge vessel with 5 mL of reagent water. Turn the sample valve back to drain, perform a manual drain, and repeat with a second flush (this is done automatically with liquid-transfer autosamplers).
- 6. After the BAKE time is complete, the system will cycle to STANDBY until the trap cools, then remain in PURGE READY for the next sample.



#### **Needle Sparging**

**Note:** The following instructions are only guidelines. Actual methodology may require different parameters (weights, volume, etc.) than those stated.

Purge-and-trap analysis using a needle sparger is similar to that using a frit sparger, with two main differences. First, the purging efficiency of a needle sparging apparatus is generally lower than that for frit (or dispersed bubble) sparging. This can be overcome by increasing the purge time, elevating the sample temperature during sparging, or a combination of both. The other difference is the lack of automatic sample draining at the end of the analysis. Therefore, the sparge vessel must be drained manually at the end of each cycle. Remove the vessel and empty the spent sample, then either clean the vessel or install a fresh sparge vessel.

The next two sections describe liquid sample and solid sample analyses using a needle sparger on the Model 4560.

#### **Needle Sparging Liquid Samples**

- 1. Disable the Model 4560 drain by pressing [2nd] [TIME]. Select states, flows, and desorb, then turn the drain off (refer to Figure 4.9).
- 2. Follow the earlier instructions for proper syringe sample loading under *Syringe Sample Loading*.
- 3. Attach the syringe to the Model 4560 sample injection valve syringe port.
- 4. Turn the sample valve handle to the **fill** position.
- 5. Inject the sample into the sparge vessel and return the valve handle to the **drain** position (no draining will occur).
- 6. Press the [START] button to begin the analysis. The sample will purge for the set point time and cycle through the states according to the set point values.
- 7. After the Model 4560 cycles to BAKE, loosen the lower sparge nut and slide the sparge vessel off.
- 8. Clean and rinse the sparge vessel before re-installation, or install a cleaned and baked sparge vessel and tighten the sparge nut for a leak-free seal.
- 9. When the Model 4560 cycles to PURGE READY, proceed with the next sample.

#### **Needle Sparging Solid or Sludge Samples**



- 1. Weigh a clean, dry sparge vessel on an analytical balance and record the mass.
- 2. According to the current method of analysis, weigh a known sample amount into the sparge vessel. A 1–5 g sample is typical, depending on expected analyte concentration.
- 3. Install the sparge vessel with sample on the Model 4560 with the sparge vessel neck inserted properly into the sparge nut. Tighten the sparge nut for a leak-free seal.
- 4. If performing a heated sparge, set the Infra-Sparge Sample Heater to the desired set point.
- 5. Fill a 5-mL syringe with volatile-free water (see *Syringe Sampling Loading* earlier in this chapter).
- 6. Add any desired internal standard to the volatile-free water using a 10  $\mu$ L syringe.
- 7. Attach the syringe to the Model 4560 sample syringe port.
- 8. Turn the sample valve handle to the **fill** position (see Figure 4.9).
- 9. Inject the reagent water into the sparge vessel and return the valve handle to the **drain** position (no draining will occur).
- 10. Press [START] to begin the analysis sequence. The sample will purge for the set point time and cycle through the states according to set point values.
- 11. After the Model 4560 cycles to BAKE, loosen the sparge nut and slide the sparge vessel off.
- 12. Clean, rinse, and dry the sparge vessel before weighing the next sample, or install a cleaned and baked sparge vessel and tighten the sparge nut for a leak-free seal.
- 13. When the Model 4560 cycles to PURGE READY, proceed with the next sample.

## Autosampling

For specific instructions on operating and programming the Model 4560 with autosamplers, refer to the appropriate autosampler operator's manual.



## **Model 4560 Key Descriptions**

This section gives detailed descriptions of all Model 4560 keys. Use this section as a key-command reference guide and for more detailed Model 4560 configurations.

#### **Temperature Keys**

The Model 4560 has eight temperature zones. To set any of the following temperatures, press the appropriate keys (listed in bold print) to display the setting, enter the desired temperature (within the designated range), then press [ENTER]. The default temperature settings are listed in Table 1.

**Purge Temp** (set from ambient to 300°C) indicates the trap temperature during PURGE. The default setting is 20°C.

**Note:** The Purge Temp should always be set on 20°C for optimal performance.

**Desorb** (**DSRB**) **Temp** (set from ambient to 300°C) indicates the trap temperature during DESORB. The default setting is 180°C. Pressing [DESORB TEMP] twice consecu**tively** shows desorb preheat and preheat temperature values.

Table 1. Temperature Default Settings

<b>Temperature Defaults</b>		
Purge Temp	20°C	
Desorb Temp	180°C	
Bake Temp	180°C	
Valve Temp	100°C	
Xfer Temp	100°C	
Sample Temp	20°C	
Spl Inlet	40°C	
Ext Heater	100°C OFF	

**Bake Temp** (set from ambient to 300°C) indicates the trap temperature during BAKE. The default setting is 180°C.

Valve Temp (set from ambient to 300°C) indicates the valve oven temperature at all times during the cycle. The default setting is 100°C.

**Transfer (Xfer) Temp** (set from ambient to 200°C) indicates the transfer line temperature at all times during the cycle. The default setting is 100°C.

Sample (SPL) Temp (set from ambient to 100°C) indicates the sample's actual temperature (if a sample heater is present). The set sample temperature is maintained in all states except STANDBY, PURGE READY, and PREPURGE, and can be optionally heated during BAKE. This zone controls the optional sample heater. The sample temperature defaults to 20°C if this zone is on.

- 1. If an Infra-Sparge Sample Heater is present on the Model 4560, pressing [SPL Temp] **twice consecutively** brings up the sparger size menu (5 mL or 25 mL). Selecting the appropriate sparger size ensures  $\pm 1$  °C temperature control for either sample size. Toggle between sample sizes by pressing [ON]/[OFF]; pressing [ENTER] is not necessary to verify entry.
- 2. Press [SPL Temp] three times consecutively to select or deselect sparger heating during BAKE. Heating may result in more effective glassware cleanup and reduced carryover. Select ON to initiate sample heating during BAKE.



**CAUTION:** Selecting 25 mL when a 5-mL sample is being heated can lead to temperature overshoot.





**CAUTION:** The 4560 CPU terminates power to all heated zones when an "overtemp" is detected.



#### **CAUTION:**

If warning beeps are heard for overtemperature status, correct the situation before attempting further operation.

Water Management indicates the temperature of the water management zone at the Model 4560's trap inlet during PURGE, DESORB, and BAKE. During PURGE, the zone defaults to 100°C to ensure a consistent temperature pathway onto the cool trap (e.g., equivalent to the valve temperature).

During DESORB, the zone defaults to 0°C as a cooling set point for controlled condensation. The Model 4560 advances to DESORB when a <1°C change occurs during a pre-defined time interval (e.g., maximum cooldown is achieved). The temperature of this zone may be raised to optimize polar recovery/water removal trade-offs as necessary.

During BAKE, the zone defaults to 240°C. This temperature effectively bakes all water removed during desorption regardless of sample temperature.

Water-management baking may require a bake time set point of at least 7 minutes to prevent incomplete system cleanup. The allowable temperature range during BAKE is 200-240°C.

**SPL Inlet** [2nd Function of **Valve Temp**] (set from ambient to 200°C) indicates the sparge mount temperature. The default setting is 40°C. This will only be present if the sparge mount heater is installed.

Ext Heater [2nd Function of Xfer Temp] (set from ambient to 200°C) indicates the discrete multisampler valve and transfer line temperatures, or the temperature of any other 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.

**Note:** If a heated zone is over its prescribed set temperature (or has an open thermocouple), a series of four warning beeps will be heard and an "Overtemperature Detected" message will display. After approximately one minute, the Model 4560 may display which heated zone is showing over-temperature status. Correct the situation before attempting further operation.

## **Time Keys**

Seven time functions are definable on the Model 4560. To set any of the following times, press the appropriate key (identified in bold print) to display the setting, enter the desired time (0-999.99 min), and press [ENTER]. The default time defaults are listed in Table 2.

**Purge Time** indicates the duration of PURGE. The default setting is 11 min.

**Desorb** (**DSRB**) **Time** indicates the duration of DESORB. The default setting is 4 min.

**Bake Time** indicates the duration of BAKE. The default setting is 20 min.

Table 2. Time Default Settings

Time Key Defaults		
Purge Time	11 min	
Desorb Time	4 min	
Bake Time	20 min	
Prepurge	0 min	
Preheat	0 min	
Dry Purge	0 min	



**Prepurge (PRE-PRG)** [2nd Function of **Purge Time**] indicates duration of PREPURGE. The default setting is 0 min.

**Preheat** [2nd Function of **Desorb Time**] indicates duration of PREHEAT. The default setting is 0 min.

**Dry Purge (DRY-PRG)** [2nd Function of **Bake Time**] indicates duration and elapsed time of DRY PURGE. The default is 0 min. Dry Purge is not often used due to Cyclone Water Management.

**Time** • when pressed **once**, the duration of the current run is displayed.

- when pressed **twice** consecutively, the elapsed time of the current run is displayed.
- when pressed **three** consecutive times, a timer is displayed to aid in setting flow. To start and stop the timer, press [ENTER]. Reset the timer by pressing [CLEAR].

From any state, press [CLEAR] to return to the main menu, then press [TIME] to redisplay the currently running Model 4560 state.

#### **Number Keys**

Set temperatures and times with the Model 4560 number keys, 0–9 (see Figure 4.8).

### **Action Keys**

**The [ENTER] key** confirms numeric entry. Pressing [ENTER] verifies the entered value. If the value is not acceptable, promptly enter another value.

**An asterisk** (\*) flashes as a new value is entered, indicating an entry in progress. If [ENTER] is not pressed within five seconds after entering a new value, the previous value will be retained.

**Press the [START] key to begin a run** or to continue an interrupted run (see [HOLD] below). If a sequence is entered and sequencing is on, [START] is also used to begin the sequence.

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

**Note:** Only the time is stopped; the unit will continue executing the current state (e.g., PURGE).

**Abort a run and return to STANDBY** by pressing the maroon key [2nd], then [ON]. Press [ENTER] to verify the selection.



#### **CAUTION:**

Aborting the run (returning to STANDBY) this way will not drain the sample. With a sample transfer multisampler, the danger of loading the next sample over the previous one exists (see Manual Drain).



**Advance to BAKE** automatically from any state by pressing the maroon key [2nd], then [OFF]. Press [ENTER] to verify the selection. Advancing to BAKE will abort any run in progress.

Manual Drain (2nd Function of [CLEAR]) drains any sample in the sparge vessel (if the sample valve is set to drain) for 0.50 min, then advances to BAKE for the duration of BAKE time. If the Model 4560 is running, elapsed time must be stopped [HOLD] before draining is allowed. Press [ENTER] after [2nd] [CLEAR] to verify the drain command.

When not in a run state, press the **[ON]/[OFF] keys** to manually advance or reverse through the enabled states with no elapsed time running. [ON]/[OFF] keys also perform the following:

a. Enable or disable a heated zone.

*Example:* If [VALVE TEMP] is pressed, the following will be displayed:

Valve Oven 100°C Valve Actual 99°C

If [OFF] is pressed, the following will be displayed:

Valve Oven 100 OFF Valve Actual 96°C

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

b. Select between options.

*Example:* If the 2nd function key (the maroon key), then [TIME] are pressed, the Configuration menu is displayed:

Configure [ON]/[OFF]
STATES OPTIONS

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

If [OFF] is pressed, the following will be displayed:

Configure [ON]/[OFF]
STATES OPTIONS



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

c. Enable or disable an option or state. Continuing the above example, after pressing [ENTER], the following will be displayed if this option is present:

Operate DPM OFF

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

Operate DPM ON

**The [CLEAR] key**, if pressed with no set point entry in progress, displays Model 4560 readiness. The [CLEAR] key is also used during set point entry (before pressing

[ENTER]) to erase an entry in progress. When [CLEAR] is pressed (if elapsed time is not running), parameters "Not Ready" and turned-off temperature zones scroll indefinitely on the display. If all parameters are on and "Ready," a "System Ready" message displays (see Figure 4.8). As the purge-and-trap cycle runs, the display shows the current state, time, and trap temperature. If another key is pressed, the Model 4560 reverts back to the current state. Press [CLEAR] to exit the menu, then press [TIME] to redisplay the current state.

## **Function Keys**

**[F1]** Manual 6-port valve Rotation [2nd] [1]

The [F1] key manually rotates the 6-port valve when no elapsed time is running.

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

[F1] manually places the Model 4560 trap in-line with the GC (Trap to GC ON) or out-of-line with the GC (Trap to GC OFF). Manually rotate the valve by pressing the [ON]/[OFF] keys (the rotation can be heard). If the trap temperature is above 50°C when attempting manual valve rotation, the following message will appear:

Cool Trap First Before Rotate Valve

This is a safety precaution, preventing the absence of flow through a heated trap if performed in certain states, and subsequent trapping sorbent damage.



#### **[F2]** Auxiliary Thermocouple [2nd] [2]

The auxiliary thermocouple signals the Model 4560 to advance to DESORB at a settable temperature if the normal contact closures signaling "GC Ready" to the Model 4560 are not available.

Pressing [2nd] [2] displays the following:

Auxiliary 25 OFF Start 0°C

The first line displays the temperature reading for the auxiliary thermocouple input. If the auxiliary temperature zone is turned on, the Model 4560 will advance to DESORB from DESORB READY when the temperature read by the auxiliary thermocouple is at or below the temperature entered on the second line. Auxiliary should normally be off when the GC Ready contact closure is available from the GC. A Type K auxiliary oven thermocouple (Part #169749) is available from OI Analytical, if needed.

**[F3]** See "Linking Multiple Method Files for an Autosampler Run" in this chapter.

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

The control (valve) manifold is the aluminum block in the Model 4560 left bay that heats the Purge, Dry-Purge, and Bake Valves to a preset 50°C and directs flow between these valves. This zone should always be on for operation and **never** heated to above 50°C. Improper heating may cause self-test failure; monitor by pressing [2nd] [4]. For any problems with this heated zone, call the OI Analytical Technical Support Toll-Free Hotline at 1-800-336-1911.

- [F6] GC ready line [2nd] [6]. GC Ready should be set to NORMAL when using an HP 5890 GC and INVERTED when using an HP 6890, Shimadzu, or Varian GC.
- [F9] Self-test results. This display lists any errors found during the Model 4560 self-test (see "The Model 4560 Self-Test" earlier in this chapter).

Unused function keys, when pressed, return to the menu previously in use.



CAUTION:

The control manifold zone should always be on for operation and never heated to above 50°C.



#### **Autosampling Keys**

**The [SPL] key**, pressed once, brings up a variety of displays, depending on instrument configuration. If no autosamplers are present or configured, the Model 4560 displays that none are enabled. Otherwise, the autosampler start/end positions display for entry and the total/current autosampler positions display for verification.

- **Start** defines the first analysis position.
- **End** defines the last analysis position.
- **Total** verifies the total number of samples to be analyzed based on entered Start/End values.
- **Current** verifies the current position of a connected autosampler.

**Note:** Start/End positions, as well as all configuration parameters, are stored in each method.

- Pressing the [SPL] key **twice** consecutively displays a cycle counter that may be used to validate completed purge-and-trap cycles during sample sequences. The cycle counter may be reset by pressing [0].
- When the 4551 Vial Autosampler is enabled, pressing the [SPL] key **three times** consecutively displays a menu for sample transfer location. When 4551/DPM-16 autosamplers are used in tandem, select <u>DPM-16</u> as the proper location by pressing on/off, otherwise, select <u>4560</u> as the default location.

**[REPL]** (2nd Function of **[SPL]**) indicates the number of replicates per sample. This key can set up to 98 replicates of a single sample. Normally, with discrete multisamplers, # replicates = 1. Current replicate is used as a counter. Setting this value to 99 will result in infinite cycling of the Model 4560.

## **Configuration Key**

[Config] (2nd Function of [TIME]) identifies and configures Options for the Model 4560 (e.g., connected instruments) *or* defines **States** to override the **Option** configuration. Use [ON]/[OFF] keys to select between **States** or **Options** and press [ENTER].

**Options** auto-configures the Model 4560 to accommodate external OI Analytical devices such as multisamplers or other instruments. Pressing [ENTER] scrolls through each configuration option; [ON]/[OFF] enables or disables the option. For specific instrument configuration instructions, refer to the appropriate Operator's Manual.



Some options have more extensive configurations accessed by pressing [1].

#### OPERATE 4551 [1] = CONFIGURE ON

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

- **Inputs** enables or disables PURGE READY and/or DESORB READY input requirements.
- Outputs defines outputs given by the Model 4560 at PURGE, DESORB, and the start of BAKE.
- **Flows** allows the choice of whether to drain the sample during DESORB or purge the sample during BAKE.

All Configuration selections are included in saved methods, but not in prestored Method 502.2/524.2, as described in the next section.

#### **Other Keystrokes**

#### Keyboard Lock [CLEAR] [.] [-]

To ensure that no temperatures or times are changed on the Model 4560, lock the keyboard by pressing [CLEAR] [.] [-], 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, then select [OFF].

#### **Group ID** [CLEAR] [.]

Several Model 4560s can link together for RS-232-C communication using the Datalink Port on the CPU Card. Several Model 4560s can then be fully controlled with OI Analytical's PATTI II Software. Each Model 4560 on the same network must have a unique group ID.

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

# **Notes**





# Chapter 5 Maintenance

## Changing the Trap

After periods of extended use, a trap may begin to degrade, causing loss of performance. Symptoms include loss of sensitivity to selected components in a standard run and increased pressure to maintain 40 mL/min flow during PURGE. When a trap is suspected of failing, replace it. Each new trap should be conditioned at least one hour at 200°C under helium flow before use. To condition a new trap, manually place the system in BAKE and set the Bake Temp to 200°C. Also keep a "reference" trap sealed and on hand, to test system performance, if necessary.

- 1. Turn the Model 4560 off and unplug the unit when changing the trap.
- 2. Open the trap access door located in the Model 4560's back left cover. A safety switch automatically cuts trap power when the door is opened if the Model 4560 has not been turned off.
- 3. Unplug the thermocouple.
- 4. Press both sides of the stainless steel housing covering the trap and remove the housing by lifting upward.
- 5. Loosen both trap fittings with a 3/8" wrench and remove the trap. It is not necessary to use a back-up wrench on the bulkhead when loosening the fittings.
- 6. Install the new trap with the outlet (designated by an "O" on the trap nut)
  - connected to the bulkhead closest to the Model 4560's center wall. The inlet will state the trap type on the nut (see Figure 5.1).
- 7. Finger-tighten the trap plus  $\frac{1}{2}$  of a turn.
- 8. Plug in the thermocouple.
- 9. Replace the stainless steel trap cover and close the trap access door.
- 10. Leak-check the connection from the PURGE state.

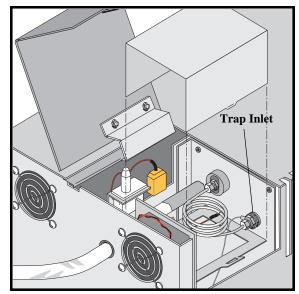


Figure 5.1. Changing the Trap



## CAUTION:

Identifying the trap inlet for blank traps, "#0," which have been custom packed, may be difficult. The trap should always be installed so the thermocouple connection is attached to the lowermost coil on the trap.



# CAUTION: The 18-mm knurled nut may be hot!

## **Cleaning/Changing the Sparger**

The Model 4560 accommodates either frit or needle-style spargers (5 or 25 mL) with minimal hardware changes. To clean a sparger, rinse thoroughly several times with ultrapure water and oven bake following normal laboratory procedures. Only use solvents when water does not clean sufficiently. Use a solvent that will not be detected by your detector.

#### **Installing a New Frit Sparger**

- 1. Loosen the top nut of the brass fitting on the purge gas inlet arm of the sparger.
- 2. Finger-loosen the 18-mm knurled nut securing the frit sparger to the bottom of the sparge mount block.
- 3. Lower the sparger until it clears the purge needle.
- 4. Place the new sparger over the purge needle and insert the sparger neck into the 18-mm fitting.
- 5. Insert the ½16" stainless steel purge gas line into the brass fitting on the purge inlet arm of the sparger.
- 6. Tighten both fittings, securing the sparger, then leak-check the connection.

### **Converting to a Needle-Style Vessel**

- 1. Remove the frit sparger as described above.
- 2. Disconnect the frit-style purge gas line (Part #227652) from the front of the Model 4560.
- 3. Replace it with the needle-style purge gas line (Part #227389) and leak-check the fitting.
- 4. Route the purge gas line through the back left cover slot of the sparge vessel door to the black sample injection valve normally used for direct injection.
- 5. Loosen the sample injection valve from the bracket, rotate the valve 90° clockwise, and retighten.
- 6. Remove the Luer-lok injection adapter from its old position and thread the Luer fitting into the frontmost port on the sample injection valve.
- 7. Connect the needle-style purge gas line to the 9 o'clock position on the sample injection valve and finger-tighten.
- 8. Slide the appropriate needle sparger over the Purge Needle into the 18-mm knurled nut and finger-tighten. Leak-check the connection.

# 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.

- 1. Remove the filter by turning it counterclockwise with a 3/8" wrench.
- 2. Clean the filter by rinsing it with methanol and drying with ultrapure nitrogen or helium.
- 3. Optionally bake the filter at 100°–150°C for 30 minutes
- 4. Replace the filter by finger-tightening it plus 1/4-1/2 of a turn.
- 5. If foaming samples are common, it is recommended that one filter (Part #209254) be kept baking in the GC oven at all times for quick replacement.

### **Interior Maintenance**

The following maintenance procedures involve changing/upgrading specific Model 4560 interior components. Only the following outlined procedures are authorized by OI Analytical for customer maintenance. Any other procedures involving the interior components of the Model 4560 should be performed by OI Analytical trained technical support personnel only.

### **Upgrading Firmware**

Upgrade the Model 4560 firmware by replacing the EPROM memory chips (Part #228106) on the CPU Card. To replace the EPROM memory chips, follow these instructions.

- 1. Turn the Model 4560 power off.
- 2. Remove the CPU card from the unit (see Figure 2.2 in Chapter 2).
- 3. Remove the old EPROM memory chips (U17 and U18) by gently prying them up with an extractor or a small flathead screwdriver if an extractor is not available.
- 4. When inserting the chip pins into the appropriate holes in the CPU Card, make sure the pins are not bent.
- 5. Pay particular attention to the notch positions at the tops of the chips. The notches must "point to" the top of the CPU Card. 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." Completely insert the chips.
- 6. Turn on the Model 4560. If the Model 4560 does not have a sparge mount heater, let the Model 4560 run through the self-test.





#### CAUTION:

OI Analytical does not authorize customer removal of the Model 4560 main cover. If a fuse located under the cover needs replacement, contact OI Analytical's technical support for assistance.

- 7. If the Model 4560 has a sparge mount heater (serial number beginning with "M" or higher):
  - Abort the self-test by pressing [CLEAR] several times.
  - Press [SPL TEMP] [OFF] to set the SPL TEMP off.
  - Turn the Model 4560 off and then back on. Let the Model 4560 run through the self-test.

**Note:** If SPL TEMP is not set to OFF, the Model 4560 will fail the self-test.

#### **Replacing the Battery**

The CPU Card's lithium battery (Part #211953) should function properly for several years. The Model 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:

- 1. Turn the Model 4560 power off.
- 2. Remove the CPU Card from the Model 4560.
- 3. To remove the old battery, gently pry it up with a small flathead screwdriver.
- 4. The "+" lead (as marked on the battery's side) should be inserted into the socket also marked with a "+." After confirming proper battery orientation, insert both leads into their corresponding sockets.

### **Replacing Fuses**

Fuses protect the electronics from failing if a short circuit condition occurs with one of the Model 4560 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, remove the cord so the Power Entry Module cover can be removed using a screwdriver. Slide out the fuse holder and replace the fuse.

Fuses are also located under the blue cover on the AC/IO Board. These may be replaced by OI Analytical trained technical support personnel only. If a fuse is blown, attempt to find the condition that caused the fuse to open before replacing it. The fuses are labeled so that the heated zone that caused the failure can be inspected before the fuse is replaced.

# **Model 4560 Diagnostic Tools**



#### Purge Flow Controller/Pressure Gauge

The Model 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 trap restriction variations. A pressure gauge is connected to the flow controller output to monitor the gas pressure required to maintain the set flow. In PURGE, the purge gas flows sequentially through:

- Purge Valve
- Purge Line
- Sparge Vessel
- Valve Oven Lines
- Trap
- · Control Manifold
- Bake Valve, to vent

Total restriction caused by these components generates a backpressure of about 6–11 psi to maintain 40 mL/min purge flow, depending upon the trapping sorbents used. If a pressure above 11 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 possible. First, check the trap connections to their bulkhead fittings and the sparger neck seal. To aid in locating a leak of this type, plug the barbed fitting where purge flow is measured. When 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 easier.

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

•	STANDBY/PURGE READY	20 psi
•	PURGE	6–11 psi
•	DRY PURGE	2–3 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 does not flow during states where 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 "system pressure" is regulated to 20 psi by the internal pressure regulator.



The trap is not in-line with the purge gas during states where 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 sparge 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 the GC or Model 4560 is a flow controller (with Internal Carrier Flow Option) for regulating the carrier gas flow through the column. If this flow controller has an outlet pressure gauge to measure column head pressure, then it can 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, requiring 30–50 psi at the column head. The carrier flow rate for 0.53 mm I.D. capillary-column purge-and-trap analysis is typically 7–10 mL/min, requiring only 5–20 psi to maintain. As the column temperature increases, more pressure is required to maintain the set flow rate. When the Model 4560 connects to the GC, the flow from the controller goes through the Model 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 pressure increase with the temperature ramp used for analysis. If a higher pressure than normal is later observed, a restriction in the 6-port valve, transfer line, or the injector is possible. If a lower column pressure than normal is observed, a leak in the components between the flow controller and the column has developed. In this case, the injector septum on the GC is most suspect. If a shift in retention times of each of the components is observed, examine the carrier pressure gauge for abnormal pressure. In this case, if nothing is wrong with capillary carrier pressure, the flow controller may be unsuitable for flow rates less than 10-20 mL/min.

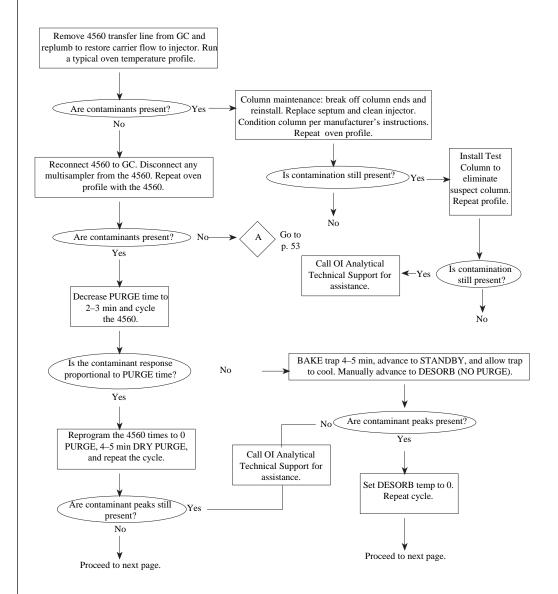


# **Chapter 6 Troubleshooting**

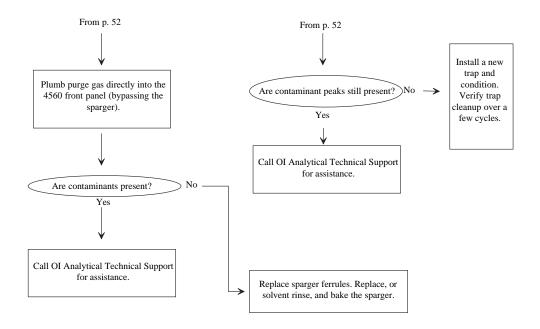
The following tree diagrams list problems that might occur during normal operation of the Model 4560 Sample Concentrator, along with possible solutions. Any maintenance that involves removing the Model 4560's main cover should be performed by OI Analytical trained technical support personnel only. If a problem still exists after reviewing the following diagrams, contact OI Analytical's Technical Support Department at (800) 336-1911 or (979) 690-1711.

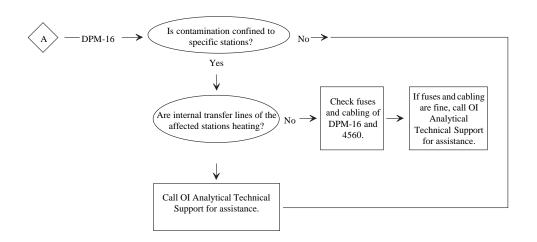
#### **Contaminant Peaks**





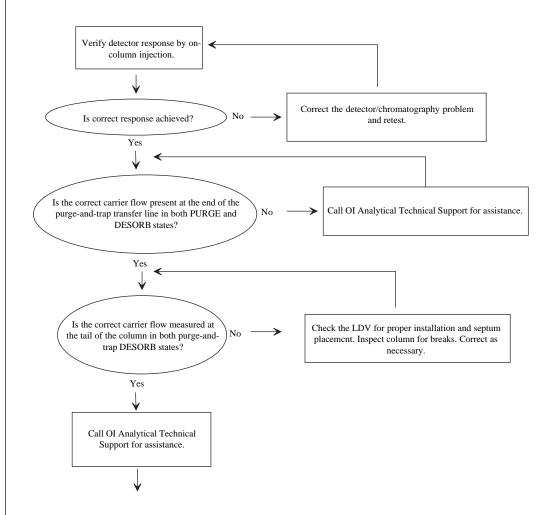




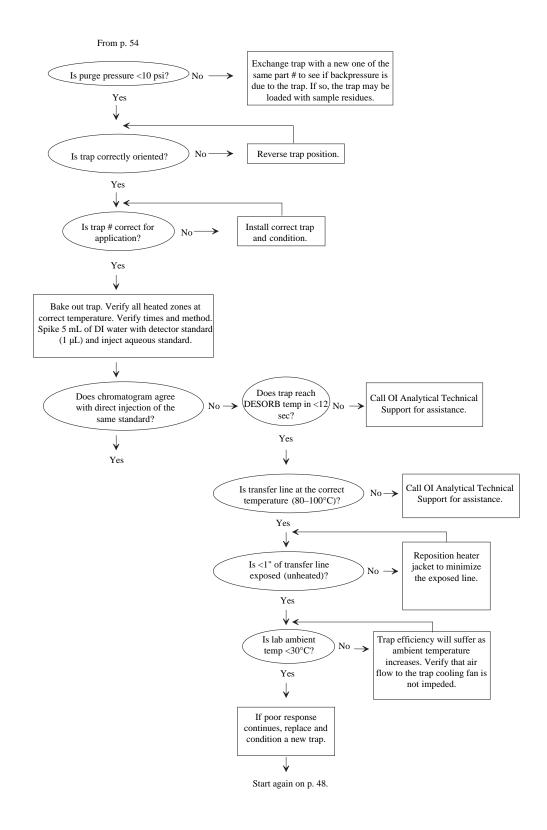






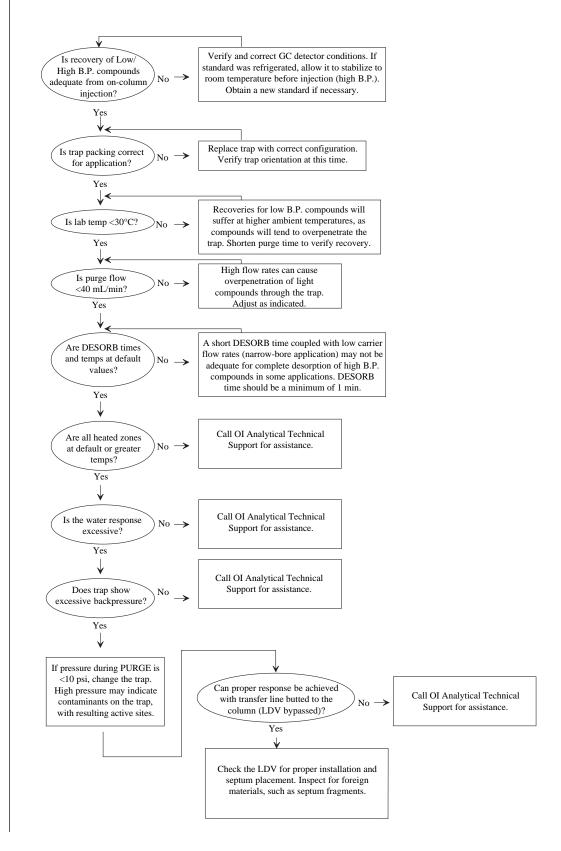






## Poor Recovery of Low/High Boiling Point Compounds







# **Chapter 7 Replacement Parts**

This chapter lists the order numbers for replacement parts and support items for the Model 4560 and its associated options. Replacement parts considered as expendable (XPND) are marked with an asterisk. (Expendable components should be replaced regularly or are easily broken or deformed.) A supply of XPND parts should be kept on hand. Units of measure (U/M) are also given. If a part is shown in a drawing, the figure number is listed under "Fig." This list is provided for ordering purposes only. Any internal parts should be installed by OI trained service personnel only.

# **Model 4560 Sample Concentrator Parts**

Part Name	Part #	<u>U/M</u>	XPND
Boards			
I/O Board w/external heater	. 298786	ea	
CPU - Model 4560	. 228098	ea	
<b>Communication Cables</b>			
Model 4560 to CFM	. 226548	ea	
Model 4560 to DPM-16, MPM-16, or MHC-16	. 226605	ea	
Model 4560 to PATTI II (RS-232-C)	. 226621	ea	
Model 4560 to any Handshake Pigtail	. 226530	ea	
MHC-16 (Rev A) to DPM-16 or MPM-16	. 192443	ea	
MHC-16 (Rev B) to DPM-16 or MPM-16	. 229450	ea	
Handshake Pigtail - HP 5890	. 185868	ea	
Handshake Pigtail - Varian 3400	. 188409	ea	
MSD Interface Cables (Replace #185868 wl	hen MSD is	present	:)
Handshake Pigtail HP 5971 or 5972 (DOS/UNIX)	. 209874	ea	
Handshake Pigtail HP 5971 (PASCAL)	. 209866	ea	
Handshake Pigtail HP 5970 (RTE-A)	. 209882	ea	
Fittings and Ferrules			
Ferrule - Teflon 1/16 Tube	. 196220	5/pk	*
Ferrule - Teflon 6 mm Tube (Sparger Inlet Arm)		ea	*
Ferrule - Teflon 18 mm Tube (Spgr. Mount)	. 224352	10/pk	*
Fitting - Adapt KEL-F F-Luer 10-32	. 224584	ea	*
Fitting - Nut SS 1/16 Male Knurled	. 217240	ea	*
Fitting - Union Br 1/4-1/16 Male		ea	*
Software Control			
WinTrap Plus	. 307488	ea	
Spargers (Sparge Vessels)			
Needle - Disposable 10 mL, 18 mm Neck O.D	100521 1	25/case	*
Frit - 5 mL, 18 mm Neck O.D. Swage Inlet			*
1711 - 3 mL, 16 mm Neck O.D. Swage miet	. 207013	ea	



<u>Part Name</u>	Part #	<u>U/M</u>	<u>XPND</u>
Frit - 25 mL, 18 mm Neck O.D. Swage Inlet		ea	*
Needle - 5 mL, 18 mm Neck O.D. Swage Inlet		ea	*
Needle - 25 mL, 18 mm O.D. Swage Inlet	225631	ea	*
Sparger Kits (includes hardware)			
Needle Sparge Hardware Kit	227397	ea	
Traps and Trap Bulkhead Parts			
Trap - #0 (Blank)	228114	ea	*
Trap - #7 (Tenax only)	227348	ea	*
Trap - #8 (Tenax/Silica Gel)	227363	ea	*
Trap - #9 (Tenax/Silica Gel/Charcoal)	219972	ea	*
Trap - #10 (Tenax/Silica Gel/Carbon Mol. Sieve)	228122	ea	*
Trap - #11 (Vocarb 3000)		ea	*
Trap - #12 (BTEX) - Carbopack B/Carbopack C	258848	ea	*
<b>Tubing and Tube Assemblies</b>			
Transfer Line - SS/Silicon 48 in x 0.020 I.D	249185	ea	
Tube Assy - Purge Gas/Needle Sparger	227389	ea	
Tube Assy - Water 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	227520	ea	
Tube Assy - Purge Out/Gauge	227603	ea	
Tube Assy - Purge FC Out/Solenoid Manifold	227611	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	*
Tube Assy - Valve Solenoid/Sparge Manifold	228957	ea	
Tube Assy - Carrier Gas FC 6-Port Valve	234005	ea	
Tube Assy - Carrier Gas Inlet 6-Port Valve	233999	ea	
Tube Assy - Reg in Carrier Gas FC in 4560	227587	ea	
Tube Assy - Reg in to Purge Gas FC	227595	ea	
Tube Assy - Solenoid Manifold/Purge Valve	247700	ea	
Tubing - Copper 1/8 x 0.070 I.D. Cleaned	111427	ft	
Tubing - Urethane 1/8 x 1/16 I.D. Clear	166224	ft	
Other Model 4560 Parts			
Assy - DC Actuator	249003	ea	
Assy - Needle Spargers (Side Hole 22.9 cm)		ea	*
Assy - Sparge Filter	209254	ea	*
Battery - 3.6 V Backup		ea	*
Firmware (EPROM chips)		ea	*
Kit - Starter Support 4560		ea	
Power Cord - 110 V North America	116038	ea	
Manual, Operator's - Model 4560		ea	
Needle - Frit Spargers 22.9 cm		ea	*
Standard - Detector and P&T 100 ppm/MEOH		ea	*
Syringe - 10 μL x 2 in Needle		ea	*
Syringe - 5 mL Sample w/Luer		ea	*
Syringe - 25 mL Sample w/Luer		ea	*
Valve - Br/Ni 3-way Manifold Viton 12 V		ea	
Valve - Br/Ni Check ½ psi 10-32		ea	
Valve - KEL-F 3-Port Sample 10–32		ea	
Valve - SS 6-Port 1/16 Tee with Drive	248575	ea	



Part Name	Part #	<u>U/M</u>	<u>XPND</u>
Wrench - Sparger 1-1/8" Open-end	. 227975	ea	
Infra-Sparge Sample Heater Option	. 225649	ea	
Assembly - Sparge Door	. 230052	ea	
Assembly - Lamp Cover	. 230060	ea	
Lamp - Sample Heater	. 225656	ea	*
Thermocouple - Needle Sparger Heater	234112	ea	

# Notes





# Chapter 8 Appendix

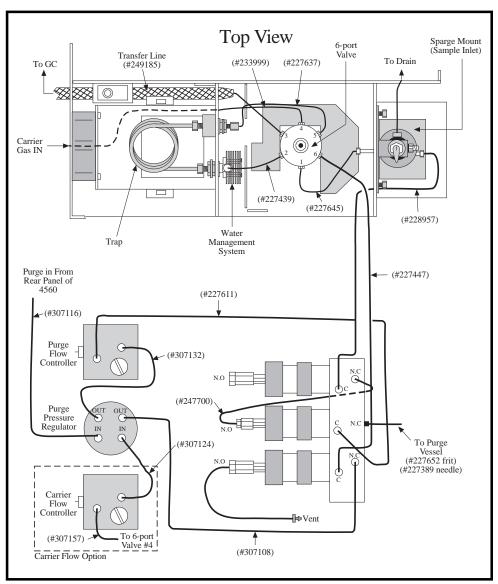


Figure 8.1. Plumbing Schematic - Model 4560s with a Serial Number Beginning with an "L" or higher, or Older Units that have had the Regulator Replaced



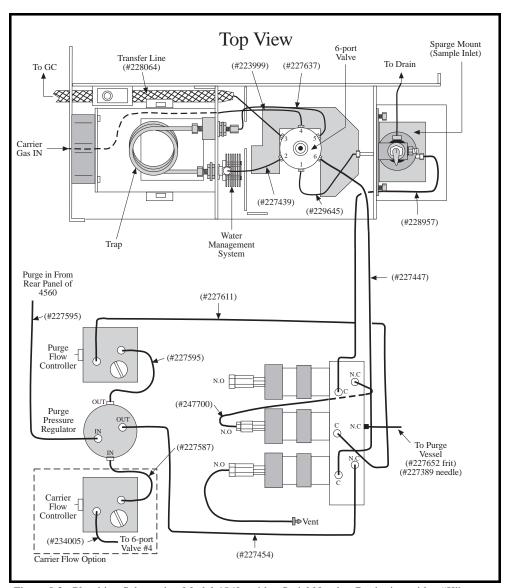


Figure 8.2. Plumbing Schematic - Model 4560s with a Serial Number Beginning with a "K" or lower



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