

Model 4430 Photoionization Detector Operator's Manual





151 Graham Road • P.O. Box 9010 • College Station, Texas 77842-9010 Telephone (979) 690-1711 • FAX (979) 690-0440 • www.oico.com



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Chapter 1 Introduction

The Model 4430 Photoionization Detector is designed to be installed in a standard detector port of an Agilent 5890A or Varian 3300/3400 gas chromatograph (GC). It is integrated into the GC by using the GC's electrometer, signal output circuit, hydrogen and makeup gas flow restrictors, detector base heater, and external event relay for the vent valve. The detector consists of an ionizing chamber, a UV lamp, and a column interface snout as an assembly mounted on the GC, and a stand-alone high voltage power supply for the lamp.

The lamp produces radiation in the UV spectrum (typically 10.0 eV) that ionizes certain molecules eluting from the GC column into the chamber. The detector can be made selective by using a lamp that outputs light at an energy level that is sufficient only to ionize components with ionization potentials below that energy level. Ions produced by the UV light are accelerated by a polarizing voltage onto a collector, and the resulting current flow is measured by an electrometer. The detector is typically used to selectively detect aromatic and olefinic hydrocarbons in the presence of alkanes and other saturated hydrocarbons.

Principle of Operation

The sample stream flows through the detector's ionization chamber where it is continuously irradiated with high energy ultraviolet light. When compounds are present that have lower ionization potentials than that of the irradiation energy they are ionized. The ions formed are collected in an electric field, producing an ion current proportional to compound mass. The ion current is amplified and output by the chromatograph's electrometer.

Features

- Custom engineered to fit specific gas chromatograph models.
- It is fully integrated into the gas chromatograph by using existing GC components and features.
- It is designed to be operated in combination with the Model 5320 Electrolytic Conductivity Detector (ELCD) or Model 4410 Flame-Ionization Detector (FID) as a dual detector set, while occupying only one detector port. In addition:
 - The ELCD and PID can be mounted and operated separately from the other.

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- Each can be operated independently of the other when the PID and the ELCD or FID are mounted together.
- The ELCD and PID can operate with or without the other when they are mounted in separate detector ports.
- A unique UV-lamp window sweep gas feature minimizes window surface contamination from column conditioning and bleed from ramping to high column temperatures. This sweep gas also prevents polymerization reactions involving certain GC eluents from occurring on the lamp window.
- Custom GC interfaces are provided for each GC model, making installation quick and simple and enhancing compatibility of components.
- Use of GC-supplied electronics enhances compatibility of components as a system.
- Mounting the PID and the ELCD reactor together enhances capillary performance by minimizing dead volume and interfacing between detectors. This unique combination also allows use of the ELCD reaction gas as a PID sweep gas and allows venting of the injection solvent and column bakeout from the PID as well as from the ELCD.
- The Model 4430 is designed to be compatible with megabore capillary column efficiencies and capacities without makeup gas.
- A 10.0 eV lamp is standard, and others are available for universal or selective response.
- An exclusive lampsaver circuit is featured to turn the lamp off, if desired, when not in use, to improve lamp life. The lamp can be automatically restarted with a manual or remote signal.
- Lamp current can be set from the front panel as one of ten settings. Lamp current is proportional to lamp output intensity.

Specifications I

Dynamic Range

>106

Linear Range

• > than 10^{6}

Sensitivity

• <40 pg (benzene)

Maximum Operating Temperature

275°C

Detector Volume

• Approximately 50 microliters

Gas Requirements

• He (99.999%)

Materials of Construction

- Inlet Glass-lined stainless steel
- Ion Chamber Gold-plated stainless steel

Solvent Vent Valve

Remotely controlled

Lamp Current

• 0–1.60 mA in 0.15 mA steps

Lampsaver Time

• 0.5–2 hrs, reset by external contact

Power

- 105–125 (±10%) VAC; 220–240 (±10%) VAC
- 48–62 Hz, Line ~50 VA
- Fuses Type AGC (fast-acting) 0.5 A

Dimensions (Lamp Power Supply)

- 5 3/4" H x 2 3/4" W x 9" D
- (14.5 cm x 6.9 cm x 23 cm)

Weight

• 5.5 lbs (3 kg)

Compliance and Safety Information

The OI Analytical Model 4430 PID meets the following International Certification when tested in typical configuration:

LVD 73/23/EEC:1974

IEC 1010-1: 1990 + A1/EN 61010-1: 1993

The Model 4430 PID also meets the following Electromagnetic Compliance Certification:

Directive 89/336/EEC: 1989

EN50082-1: 1992 IEC 801-2/EN61000-4-2 IEC 801-3/EN61000-4-3 IEC 801-4/EN61000-4-4 CISPR 11:1990/EN55011 (1991)

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The Model 4430 PID 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 4430 PID 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 4430 PID must be connected to the AC power supply mains 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

- This unit must be supplied with a grounded receptacle.
- Disconnect the AC power cord before removing any covers or working on the detector. Failure to turn off the detector for service or installation can expose the operator to high voltages and possible electrical hazards.
- Turn off the main power switch and disconnect the main power cord before using a liquid solution to locate leaks.
- 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 cannot become kinked, punctured, or otherwise damaged, and will not interfere with foot traffic.
- Do not restrict airflow on the back and/or bottom of the unit. This can cause overheating or the inability to remove heat from within the unit.

WARNING: The power cable must be disconnected from the detector controller before ANY service or maintenance is performed on the

detector.

- Maintain a static-safe area when handling all electronic parts and assemblies. Use a static-control wrist strap that is connected through a one megaohm resistor to an appropriate earth ground. Store all electrical parts and equipment in static-protective containers.
- The GC oven, inlet, and detector zones, as well as the detector, may be hot enough to cause burns. Turn off all heated zones and allow time for cooling before working on the GC or the detector.
- Wear safety glasses to prevent possible eye injury.
- Do not replace blown fuses inside the detector controller. Only trained service personnel should access the interior of the detector controller.
- 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.
- Verify that all heated areas have cooled before handling or wear adequate hand protection to prevent burns.

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 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.
- Hydrogen is extremely flammable and has been identified as an asphyxiant. This gas and the cylinder containing it should be handled and stored in a manner consistent with OSHA regulations. Open flames and easily ignited materials should not be brought in contact with hydrogen except under approved, controlled conditions by the analyst. Adequate ventilation should be maintained in areas where this gas is used and stored. Avoid prolonged exposure to high concentrations of this gas. In any application using hydrogen, turn off the supply at its source before working on the GC or the detector.
- 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 stored. The analyst should avoid prolonged exposure to high concentrations of these gases.

WARNING: Hydrogen is highly flammable and may cause an explosion if it is allowed to build up in an enclosed area, such as in the GC oven. Great care should be exercised when handling hydrogen. Leak check all gas fittings periodically and keep open flames and other sources of ignition clear of the detector.

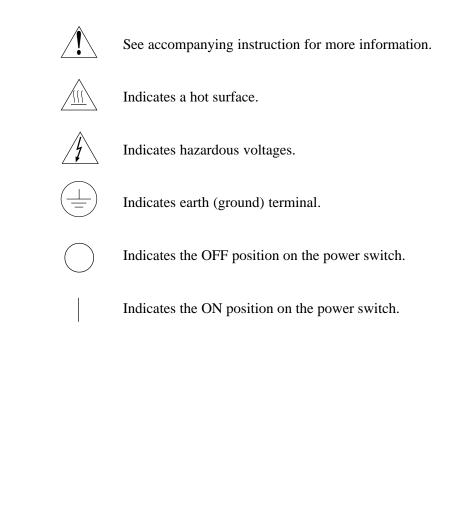
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• Oxygen has been identified as an oxidizer. This gas and the cylinder containing it should be handled in a manner consistent with OSHA regulations. Open flames and easily ignited materials should not be brought into contact with the purge gas except under approved, controlled conditions by the analyst. The operator should also avoid prolonged exposure to high concentrations of this gas.

Safety Symbols

The following symbols are located on the instrument:





Chapter 2 Description of Components

In Chapter 1, some basic concepts of Model 4430 operation as well as its features and specifications are outlined. Chapter 2 deals with the various components of the Model 4430 and their functions.

Main Detector Components

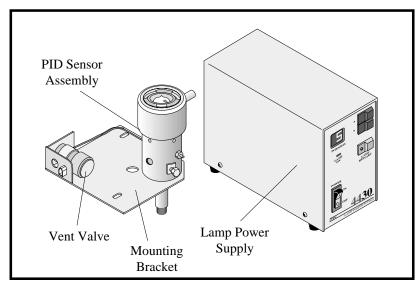


Figure 2.1. Model 4430 PID Sensor Assembly and Power Supply

Sensor assembly houses the lamp, the ionization chamber, the ion collector, and the ports for the various gas inputs. It is designed specifically to mount on an available GC detector port quickly and easily without modification of the GC.

Vent Valve allows venting of unwanted column effluents before they move into the ionization chamber. The vent valve is useful when conditioning new columns, for venting solvent peaks, and for selecting against components eluting within specific retention time windows.

Lamp Power Supply provides current by means of high voltage DC power to the UV lamp for its proper operation. Lamp current is adjustable in steps. The lamp power supply also houses the lampsaver circuit, which can be set to automatically turn the lamp on and off under its own or remote control.

Power Supply - Front Panel



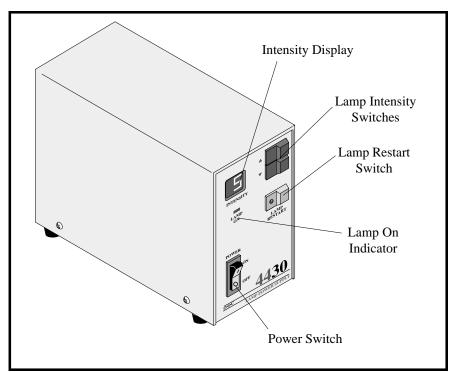


Figure 2.2. Front Panel of Model 4430 Power Supply

Power Switch activates lamp power by pressing the top of this key. Power status is indicated by a neon lamp on the power switch.

LAMP ON Indicator LED comes on when the voltage and current to the UV lamp are within its operating window, thus indicating that the lamp is on. This LAMP ON indicator will turn off in the case of an open or shorted circuit, or a failed lamp.

INTENSITY Display displays the lamp current settings in steps, from 0 to 9. The 0 setting applies no power, and each successive step increases the lamp current by 0.15 mA, with the exception of the first step which is 0.4 mA (so that the range of current that can be applied to the lamp is 0–1.60 mA). The lamp intensity is proportional to the lamp current; higher settings as shown in the INTENSITY display yield a higher detector response and baseline.

Lamp Intensity Switches are used to increment the lamp intensity up or down in steps from 0 to 9, as indicated by the INTENSITY display. The intensity setting will "roll through" 9 to 0 with continued stepping of either of the lamp intensity switches.

LAMP RESTART Switch is part of the lampsaver feature described under "Power Supply - Rear Panel" in this chapter. It is used to restart the lamp once it has been turned off by the lampsaver timer circuit. Pressing the LAMP RESTART switch restarts the lamp and the lampsaver timer.

Power Supply - Back Panel

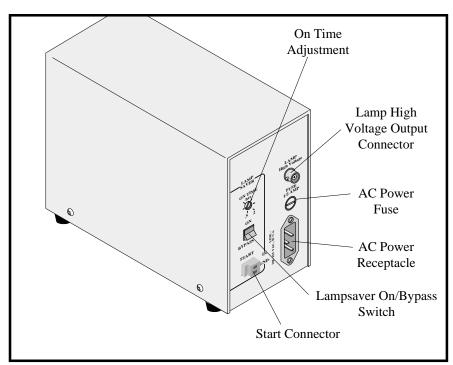


Figure 2.3. Back Panel of Model 4430 Power Supply

AC Power Fuse provides overall protection to the lamp power supply against damage due to electrical malfunction. Use a 0.5 amp fast-blow as a replacement fuse.

AC Power Receptacle receives 110 V or 220 V power, by means of a modular power cord, for operation of the lamp power supply.

LAMP High Voltage Output Connector is a BNC connector that passes the power for the lamp. Its cable connects to the BNC connector on the PID sensor.

Lampsaver ON/BYPASS Switch activates the lampsaver circuit. When this switch is set to ON, the lamp will stay on for the time controlled by its internal timer, then it will automatically turn off to extend lamp life. The time the lamp stays on is adjustable using the ON TIME adjustment. The lamp can be restarted by an external signal to the START connector using the supplied cable or by pressing the LAMP RESTART switch on the front panel. When either of these "restarts" is used, the timer restarts as well as the lamp; the lamp will remain on as long as the power supply receives another restart signal before the lampsaver times out. When this switch is set to BYPASS, the lampsaver circuit will not function, and the lamp will stay on indefinitely or until turned off manually.

ON TIME Adjustment is used to set the amount of time the lampsaver timer will run before it turns off the lamp. It is continuously adjustable between 0.5 and 2 hours.

START Connector accepts a switch or relay closure, as well as an active-low TTL, 12 VDC, or 24 VDC event signal, to restart the lamp and lampsaver timer by remote control, generally by using the GC's remote start or timed-events capabilities.



CAUTION: Voltage requirement is in the range of 105– 120 VAC, 50–60 Hz only.





CAUTION: Always match the polarity of all inputs of devices controlled by the same event signal. Typically, a single event signal is used to simultaneously restart this timer, the GC, and the data system.

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Sensor - For Agilent 5890



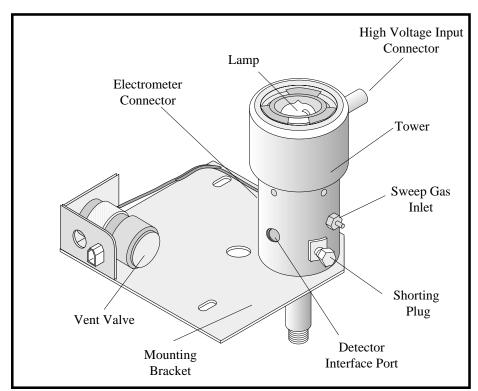


Figure 2.4. Model 4430 Sensor for the Agilent 5890

Detector Base (Not Visible) beneath the mounting plate houses the heater cartridge and temperature sensor for heating the detector. At its bottom is a snout which protrudes into the GC oven to accept the column. The snout uses a 1/4" Swagelok-type compression seal for connecting the column or a column adaptor.

Detector Interface Port can receive the reactor of a Model 5320 Electrolytic Conductivity Detector (ELCD) or a Model 4410 Flame-Ionization Detector (FID) so that the resulting dual detector set can be used with a single column. The PID is nondestructive and can operate as the "upstream" detector in the series. In this case, the PID sweep gas is used as the ELCD reaction gas or the FID fuel gas, and the vent works for both detectors.

Electrometer Connector: Through this connector passes the ion current to the electrometer installed in the GC by means of a coaxial cable. It is electrically connected to the ion collector inside the sensor.

High Voltage Input Connector: Through this BNC connector passes power for the lamp. Its cable connects to the BNC connector on the lamp power supply.

Lamp emits high energy ultraviolet light for the ionization of specific molecules. The standard lamp puts out an energy of 10.0 eV, which makes the detector selective for aromatics and other unsaturated hydrocarbons. Other lamp energies are available.



Shorting Plug holds specific components of the sensor at the ground potential, which is necessary for proper operation using the Agilent 5890 electrometer.

Sweep Gas Inlet uses a 1/16" Valco-type compression seal for the sweep gas to enter the ionization chamber. The sweep gas acts to "hide" the lamp window from the GC effluent so that its surface does not accumulate contaminants or participate in polymerization reactions of certain compounds eluting from the column.

Tower is the main body of the PID sensor. It houses the lamp, ionization chamber, and ion collector. The top half of the tower is removable for quick lamp changing.

Vent Outlet connects the base of the ionization chamber to a vent valve so that unwanted solvent, components, or column bleed can be vented instead of being detected.

Vent Valve opens on command from the GC to vent unwanted effluents as described above. When the vent valve is open, flow can be measured through its barbed outlet fitting. The flow should be adjusted to allow 35–40 mL/min to exit.

Sensor - For Varian 3300/3400

The components of the Varian 3300/3400-style sensor are identical in function to those of the Agilent 5890-style, with the exception that the shorting plug is turned into a polarizing voltage input connector.

Polarizing Voltage Input Connector: Through this connector passes the polarizing voltage provided by the GC for proper operation of the sensor with a Varian electrometer.

Other Components: Other components are identified in this section's photograph. The function of each is outlined in the previous section. The Varian-style sensor uses the heated detector plate provided by the GC to heat the detector base, so it incorporates no internal heater or temperature sensor.



Notes



Chapter 3 Installation

In Chapter 2, the names and functions of the various components of the Model 4430 were described. These components are involved in the installation of the detector onto a gas chromatograph.

The Model 4430 is custom engineered to fit neatly on an Agilent Model 5890A or a Varian Model 3300/3400 gas chromatograph. In each case, the detector uses the electrometer supplied for use with these GCs. Installation of the Model 4430 on other models of gas chromatographs is possible, but its installation and operation are not guaranteed by OI Analytical.

After opening the shipping container, unpack the instrument 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 the detector has been 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 OI Analytical Order Entry Department at (800) 336-1911 or (979) 690-1711.

Installation on an Agilent 5890

Preparing the GC

- 1. Turn off the GC power.
- 2. Remove the GC oven top, and the top right, right side, and back covers.
- 3. Remove the rear (B) detector port cover and insulation plug.
- 4. If necessary, remove the electrometer board to be used with the PID from the GC. Remove its FID interconnect tube by loosening the three mounting nuts.
- 5. Replace the interconnect tube with the electrometer input cable provided with the Model 4430.
- 6. On the GC motherboard (GC right side), disconnect the brown plug J10 and remove the clear plastic housing.



CAUTION: When inserting tubing through insulation, take precautions to prevent clogging the tube end with fibers.

Corresponding pinouts for detector A and B heaters and PRT sensors are marked on the motherboard. Refer to the GC manual for more information as needed.



CAUTION: Always match the polarity of all inputs of devices controlled by the same event signal. Typically, a single event signal is used to simultaneously restart the lampsaver timer, the GC, and the data system.

- 7. Now plug J9 is exposed. Unplug it by squeezing its two locking levers.
- 8. Unplug plugs J7 and J8.

Installing the Model 4430

- For capillary columns, a makeup gas line is supplied and should be routed to the detector. Route the line from the flow control area on the left side of the 5890, through cut outs in the plastic at the top of the instrument, and across the top of the oven to the area of the detector. Insert 4¹/₂" of line through a hole near the B-port, through the insulation, and through the underside makeup gas hole between the two detector ports in the top of the oven.
- 2. After confirming that the heater cartridge and temperature sensor are fully inserted in the detector base, orient the Model 4430 sensor in the detector port so that its detector interface port points towards the back and the detector base snout protrudes into the oven.
- 3. Insert the electrometer into its slot in the GC, while guiding the electrometer input cable into its mating receptacle on the sensor. Tighten the threaded connector, then secure the sensor mounting plate with three mounting screws.
- 4. Route the base heater cable bundle from the sensor, towards the back through the GC cable tray and to the other board on the right side of the GC.
- 5. Press the heater pins (large diameter) into the GC receptacles labeled DET B on plug J9.
- 6. Press the PRT sensor pins (small diameter) into the GC receptacles labeled DET B plug J7.
- 7. Route the vent valve power cable to the motherboard along the other cable bundles to the GC motherboard, and press the two pins into the receptacles labeled PRG B on plug J8.
- 8. Return connectors J7, J8, and J9 to their receptacles. Replace the clear plastic cover and the brown connector J10.
- 9. Install the makeup gas and column. See "Installing the Makeup Gas" and "Installing the Column" in this chapter.
- 10. Connect the sweep gas line by tightening its Valco tube nut and ferrule into its port on the sensor. Route this line to a restrictor valve in the flow control area of the GC or to the supplied restrictor that provides gas flow of approximately 35 mL/min. At 50 psi, sweep gas flows of up to 100 mL/min may be used for more efficient sweeping of the lamp with only a slight decrease in sensitivity. When used in combination with an ELCD, the sweep gas must be set at 100 mL/min.



- 11. Connect the lamp high voltage input cable to its connector on the tower, route towards the back, and then to the PID lamp supply. Connect it to the power supply connector.
- 12. Connect the power cord to the power supply and to a source of 115 VAC power.
- 13. If the lampsaver circuit is to be used, connect the cable that provides the restart signal to the START connector on the power supply.
- 14. Set the ON/BYPASS switch and the ON TIME adjustment as desired, according to their functions described in Chapter 2.
- 15. Replace all the GC covers and panels.

Installation on Varian 3300/3400

Preparing the GC

- 1. Turn off the GC power.
- 2. Remove the GC top covers.
- 3. Remove any cover from the detector port in which the Model 4430 will be mounted.
- 4. Remove the electrometer board to be used with the PID if necessary, and remove its two cables.

Installing the Model 4430

- 1. Connect the polarizing voltage cable and the electrometer input cable provided with the Model 4430 to the electrometer board in place of the two removed cables.
- 2. Ensure that the FID/TSD switch on the electrometer board is set to FID, then reinstall the board in the GC.
- 3. For capillary columns, a makeup gas line should be supplied to the detector using a 36" length of 1/16" O.D. stainless steel tubing. Route the line from a makeup gas restrictor valve in the flow control area on the left side of the GC, to the detector port, down the side-slot in the port hole, and leave 4¹/₂" extended into the GC oven.
- 4. Remove the sensor tower's top half by pressing in on its two locking pins with a pointed object and pulling the top half up. Remove the now exposed lamp and O-ring.
- 5. Orient the sensor in the detector port so that the detector interface port points towards the back and the snout protrudes into the oven.

CAUTION: Be sure that the O-ring is positioned concentric in its sealing area before placing the lamp on top of it.

- 6. Tighten the two hex-head mounting screws so that the sensor is secure in the port, then reinstall the O-ring and lamp.
- 7. Reinstall the tower's top half making sure that the two locking pins pop into their alignment holes.
- 8. Connect the electrometer input cable and the polarizing voltage cable according to the color codes on the sensors connectors.
- 9. Install the makeup gas and column. See "Installing the Makeup Gas" and "Installing the Column" in this chapter.
- 10. Connect the sweep gas line by tightening its Valco tube nut and ferrule into its port on the sensor. Route this line to a restrictor valve in the flow control area of the GC, or to the restrictor provided for gas flow in the 35 mL/min range. Sweep gas flows of up to 100 mL/min may be used for more efficient sweeping of the lamp with only a slight decrease in sensitivity. When used with an ELCD, the sweep gas must be set at 100 mL/min.
- 11. Connect the vent outlet line to the sensor and the vent valve, then mount the vent valve with its bracket onto an existing threaded hole in the oven top.
- 12. Connect the vent valve power wires to a set of 24 VDC output terminals on the GC relay board, as desired, for remote vent control.
- **Note:** See the GC manual for more information on operation of external devices such as the vent valve.
- 13. Connect the lamp high voltage input cable to its connector on the tower, route towards the back, and then to the PID lamp power supply. Connect it to the power supply connector.
- 14. Connect the power cord to the power supply and to a source of 115 VAC power.
- 15. If the lampsaver circuit is to be used, connect the cable that provides the restart signal to the START connector on the power supply.
- 16. Set the ON/BYPASS switch and the ON TIME adjustment as desired, according to their functions described in Chapter 2.
- 17. Replace all the GC covers and panels.



Installing the Makeup Gas

- 1. Remove the PID makeup gas adapter (Part #186338) from its packaging and the ¹/₄" nut (Part #169682) and ¹/₄" ferrule (Part #222794) from the PID kit (see Figure 3.1).
- 2. Slide the nut and then the ferrule over the ¹/₄" end of the makeup gas adapter. See Figure 3.4 for the orientation of the ferrule.
- 3. Remove the $\frac{1}{4}$ " plug nut from the PID inlet, from inside the GC oven.
- 4. Slide the makeup gas adapter into the PID inlet as far as it will go (see Figure 3.1).
- 5. While pushing up gently on the makeup gas adapter, tighten the ¹/₄" nut until it is secure. Orient the makeup gas inlet nut so that it points to the left.
- 6. Attach the free end of the makeup gas line, which was previously routed into the GC oven, to the makeup gas tee fitting using the supplied 1/16" nut and ferrule (Part #204693).
- 7. Leak check all gas line connections.
- 8. The gas flow requirements for the Model 4430 are provided in Table 3.1.

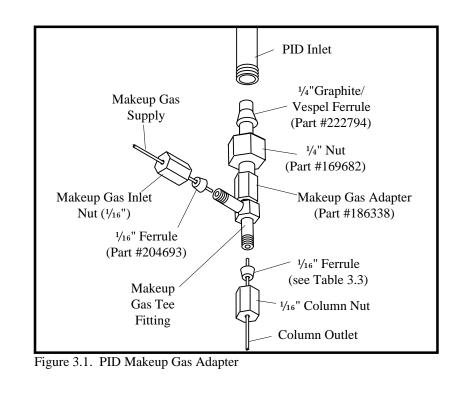




Table 3.1. Gas Supply Requirements for the Model 4430 PID

Gas Requirement	Species	Required Flow Rate
PID makeup gas PID sweep gas	N_2 or He H_2 or He	10–30 mL/min 20–40 mL/min

Installing the Column

The PID base is designed for 0.53 mm or smaller I.D. capillary columns. To install the column into the detector base:

- 1. Open the GC oven door. From inside the GC oven, remove the bottom $\frac{1}{16}$ " colum nut from the makeup gas adapter.
- 2. Slide the column nut and the appropriate ¹/₁₆" GRP/VSP ferrule onto the column, with the tapered end of the ferrule facing into the column nut. See Table 3.2 for the appropriate ferrule.

Table 3.2. Column Ferrules for Use with the PID

Base	Column	Ferrule I.D.	Material I.D.	Part No.
1/16	0.53	0.8 mm	GRP/VSP	#196105
1/16	0.32	0.5 mm	GRP/VSP	#196113
1/16	< 0.32	0.4 mm	GRP/VSP	#208330

- 3. Using a proper column cutting tool, cut a small section off the end of the column to remove any foreign particles that may have lodged in the column's open end. Check for a clean, straight cut.
- 4. With the ferrule and nut on the column, push the column up into the inlet at the base of the PID until the column outlet extends approximately 98 mm from the bottom (back) of the column nut to the end of the column. For 0.53 columns, push the column until it stops and pull back 0.5 mm. Be careful to NOT extend the column beyond 98 mm. With the other hand, finger-tighten the ¹/₁₆" nut in a clockwise direction. If the column is not secure, use a ⁹/₁₆" wrench to tighten the nut sufficiently. Do not overtighten. Pull down lightly to verify that the column is not broken.
- 5. Leak check the connection.



Chapter 4 Operation

In the last chapter, installation of the Model 4430 was described. This chapter deals with the operation of the detector. Because the detector electronics are so integrated into the existing GC components, operation of this PID closely resembles operation of the GC's standard flame-ionization detector (FID). For this reason, we recommend that you review the operation of the FID in the respective GC manual. Of particular importance is an understanding of:

- how to heat the detector base and display its temperature;
- how to turn on the detector electronics and display its output;
- how to get a signal output from the detector to a recorder or data system; and
- how to control a solenoid valve (in this case the PID vent valve), if desired, from a 24 VDC output within the GC.

Each of these aspects of detector operation is covered in the GC manual and should be understood before proceeding.

Recommended Settings for General Operation

Typical Settings with the Agilent 5890

Base Temperature

• 200°C or 20°C above the highest column temperature, 275°C maximum

Range on GC

• 2⁴

Attenuation on GC

• 2^o (does not affect output used)

Zero Offset on GC:

• 0.0

Signal Output Scale

• 0–1 volt (for integrator)

Lamp Intensity Setting

5



Sweep Gas Flow

• The sweep gas used for the PID is either H_2 or He, and the appropriate flow rate is determined by the detector configuration shown in Table 4.1.

Table 4.1. Gas Flow Rates for PID Stand-Alone and Tandem Detectors - Agilent 5890

Detector Configuration	Sweep Gas Species	Flow Rate mL/min
Model 4430 Stand-Alone	H_2 or He	20-40 mL/min
Model 4450 PID/FID	ΓH ₂	32-38 mL/min
Model 5350 PID/ELCD	H_2	100 mL/min*
Model 5390 PID/XSD	N_2 or He	20-35 mL/min
*100 mL/min or enough H_2 to give a total gas flow of at least 135–160 mL/min		

Makeup Gas Flow

• The makeup gas used for the PID is either He or N₂, and the average flow rate for stand-alone or tandem detector configurations is 20 mL/min (if column flow is less than 10 mL/min).

Typical Settings with the Varian 3300/3400

Base Temperature

• 200°C or 20°C above highest column temperature, 275°C maximum

Range on GC

• 10

Attenuation on GC

• 1 (does not affect output used)

Zero Offset on GC

• 0.0

Signal Output Scale

• 0–1 volt (for integrator)

Lamp Intensity Setting

• 5

Sweep Gas Flow

• The sweep gas used for the PID is either H_2 or He, and the appropriate flow rate is determined by the detector configuration shown in Table 4.2.



Table 4.2. Gas Flow Rates for PID Stand-Alone and Tandem Detectors - Varian 3300/3400

Detector Configuration	Sweep Gas Species	Flow Rate mL/min
Model 4430 Stand-Alone	H ₂ or He	20-40 mL/min
Model 4430 PID/4415 FID	Γ́Η,	32-38 mL/min
Model 5350 PID/ELCD	$\tilde{H_2}$	100 mL/min*
Model 5390 PID/XSD	N_2 or He	20-35 mL/min
*100 mL/min or enough H, to give a total gas flow of at least 135–160 mL/min		

Makeup Gas Flow

• The makeup gas used for the PID is either He or N₂, and the average flow rate for stand-alone or tandem detector configurations is 20 mL/min (if column flow is less than 10 mL/min).

General Operation

- 1. Verify that the column, sweep gas, and any makeup gas flows are set.
- 2. Turn on the lamp power and confirm that the lamp lights.
- 3. Note the baseline to confirm that it is at an acceptable level.
- 4. Proceed with the chromatographic analysis.



Notes



Chapter 5 Maintenance and Troubleshooting

Cleaning the Lamp Window

To prevent deposit formation on the outer PID lamp window, use the PID window polishing kit (Part #214924). Window deposits may also include residue from the O-ring, which should be cleaned with methanol and a Kimwipe.

Removing the PID Lamp

- 1. Turn off the lamp power. If an ELCD is in tandem with the PID, turn the solvent pump off.
- 2. Disconnect the BNC cable (high-voltage cable) from the PID sensor assembly.
- 3. Remove the PID tower assembly by pressing the two small retaining pins on the tower.
- 4. Gently remove the PID lamp to expose the lamp window.

Cleaning the Window

- 1. Open the lamp window polishing kit, and remove the cleaning compound and cotton swabs.
- **Note:** In the event that the window polishing kit is not available, the lamp window can also be polished using a jeweler's rouge or toothpaste and water.
- 2. Dampen the lamp window with deionized water.
- 3. Moisten a cotton swab and dip the swab tip into the cleaning compound until it is well coated.
- 4. Swab the lamp window using light pressure.
- 5. Rinse the lamp window thoroughly with deionized water and allow it to dry before reinstalling the lamp. (Blow dry the lamp window or wipe it dry using a clean towel or Kimwipe.)
- 6. Diluted acids or methanol may be used for dissolving specific contaminants if necessary.
- 7. Deposits on the window may also include residue from the lamp sealing Oring, which is best cleaned with methanol and a Kimwipe.

CAUTION: If an ELCD is in tandem with the PID, make sure the solvent pump is off. Failure to do so will damage the reaction tube, PID, and column.



- 8. Reinstall the lamp. It may be useful to replace the lamp sealing O-ring (Part #255679). Reinstall the PID tower into the base assembly, verifying that all seals, washers, and springs are reinstalled properly (see Figure 5.1).
- 9. Reconnect the BNC cable.
- 10. Once the PID is installed and operating properly, note the detector signal and record a typical baseline with zero offset on a strip chart recorder or data system. Retain a copy of a typical chromatogram to compare with future responses.

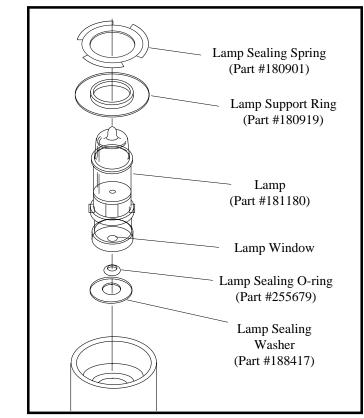


Figure 5.1. PID Lamp Assembly

Cleaning the PID Sensor—Hexane Boil Procedure

- 1. Cool the PID base to 80° C.
- 2. Cool the 5320 ELCD or 4410 FID, and remove them if they are in tandem with the PID.
- 3. Turn off the Model 4430 power supply.
- 4. Disconnect the cable from the PID sensor and remove the tower, PID lamp, Oring, and washer. Turn off the sweep gas (hydrogen) and column makeup gas (helium).

CAUTION: Do not remove the six hex nuts from the PID sensor base, as removal of these nuts could render the sensor inoperative.



CAUTION: Hexane vapors can be explosive; use extreme care when using this solvent.



- 5. Drip hexane into the center orifice of the PID sensor until the cavity is full. Raise the PID base temperature to 100°C and blot all particles and contamination with a paper towel. Repeat as many times as necessary until the paper towel no longer shows any discoloration or particles.
- 6. Reassemble the PID and restore all gas flows, then reinstall the ELCD or FID, if applicable. Raise the PID base temperature to 200°C. Monitor the PID baseline to see if the signal drops below the level prior to cleaning.
- 7. If the signal drops rapidly, allow the PID to bake out and stabilize.
- 8. If the signal is still at or near its original level, no additional cleaning may be necessary.

Additional Cleaning

- 1. If contamination is severe, the operator may want to lower the GC oven temperature to ambient (30°C) and remove the column for additional cleaning.
- 2. Mark the column at the column nut (e.g., with whiteout).
- 3. Disconnect the column, the column adapter and the makeup-gas tee.
- 4. Clean the column adapter and the makeup-gas tee externally either by sonication or soaking with a squeeze bottle of hexane.
- 5. Inspect the column end under magnification for particles or other contamination.
- 6. Cut off the column at the point where it appears clean and mark the distance from the old column end to the whiteout mark, to maintain the correct insertion depth.
- 7. Reconnect the column, column adapter, and makeup tee, then proceed with the column bake out.
- **Note:** Lamp Warranty—The PID lamp is covered under product warranty if the lamp fails to light upon installation, or if the lamp fails to function anytime within 30 days of shipment.



Notes



Chapter 6 Replacement Parts

Model 4430 Parts

Part Name	<u>Part #</u>	<u>U/M</u>
Boards		
Lamp Power Supply Front Panel Board	180497	ea
Lamp Power Supply Main Board		ea
Dump Tower Suppry Main Dourd		eu
Cables		
Lamp High Voltage Cable	181750	ea
Fittings and Ferrules	10/252	7 / 1
Adapter - Brass/Nickel 1/16 Tube x 10–32		5/pk
Assembly - Connector, SMA, PID		ea
Assembly - Heater, Reactor/PID Base		ea
Assembly - Sensor Tower		ea
Assembly - Vent Valve, 24 VDC		ea
Ferrule - 1/8 x 1/16 Graphite Tube		5/pk
Ferrule - Brass 1/16 Tube Back		5/pk
Ferrule - Brass 1/16 Tube Front		5/pk
Ferrule - Graphite/Vespel 1/4 Tube		10/pk
Ferrule - SS 1/16 Tube		5/pk
Gasket - Mounting Detector Plate		ea
Nut - Brass/Nickel 1/16 Male		5/pk
Nut - SS 1/4 Female		ea
Nut - SS 1/8 Male	112458	ea
Nut - SS 1/16 Male		5/pk
Nut - SS Outlet Restrictor	188490	ea
O-ring - Viton, Lamp		5/pk
Valve - Vent, Brass/Nickel 2-Way 24 VAC	183939	ea
Washer - Lamp Sealing	188417	ea
Tuking and Tuka Assembling		
Tubing and Tube Assemblies Tube - SS Outlet Restrictor	100107	
		ea
Tubing - Copper 1/8 x .070 I.D.		ft
Tubing - SS 1/16 x .020 I.D.		ft
Tubing - SS 1/16 x 0.030 I.D	193409	ft
Other Model 4430 Parts		
Fuse - 0.5 amp	115469	ea
Kit - Lamp Polishing		ea
Lamp - 10.0 eV		ea
Lamp Power Supply		ea
Lamp Sealing Spring		ea
r		

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Lamp Support Spring	ea
Manual - Operating and Service Procedures	ea
Power Cord - 110 VAC 116038	ea
Standard, Detector/Purge and Trap, 100 ppm MeOH 218966	ea
Vent Outlet Restrictor	ea

Parts Specific to Agilent 5890

Adapter - Makeup Gas Capillary Column	 ea
Assembly - Sensor	 ea
Cable - Analog Signal (0–1 V)	 ea
Cable - Electrometer Input	 ea
Cable - Electrometer, 14"	 ea
Cable - Electrometer, 3.7"	 ea
Cable - Vent Valve	 ea
Connector - SMA, PID Shorting Cap	 ea
Nut - Hex Nickel M3	 3/pk
Start-Up Kit	 ea
Temperature Sensor	 ea
Tube - SS Vent Outlet	 ea

Parts Specific to Varian 3400

Adapter - Makeup Gas Capillary Column to 1/4"	6346 ea
Assembly - Sensor	3954 ea
Bracket - Sensor Mounting	4853 ea
Bracket - Vent Valve Mounting 182	2923 ea
Cable - Analog Signal (0–1 V) 18	6668 ea
Cable - Electrometer Input18	1859 ea
Cable - Polarizing Voltage	4036 ea
Cable - Vent Valve	6650 ea