Model 5360A Halogen Specific Detector (XSD™) Operator's Manual for the Agilent 7890 or 6890 Gas Chromatograph

O·I·Analytical

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Limited Warranty

OI Analytical warrants each Model 5360A Halogen Specific Detector (XSD) 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.

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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 OI Analytical Model 5360A Halogen Specific Detector's (XSD) simple design provides enhanced reliability and reproducibility and significantly reduces maintenance costs. Unlike other halogen selective detectors, such as the electron capture detector (ECD) and the electrolytic conductivity detector (ELCD), the XSD contains no radioactive sources and does not use organic solvents.

The XSD quantifies halogenated compounds eluting from a gas chromatograph's (GC) capillary column. The XSD detects in the subpicogram to microgram range.

The XSD installs in a standard detector port of the Agilent® Model 6890 GC. It can be installed either as a stand-alone detector or in tandem with the Model 4430 Photoionization Detector (PID).

A venting option can be added that is controlled by the GC's event relay. If the venting option is used, air flow must pass through an in-line flow controller, such as a needle valve or a mass flow controller. Use either a manual or electronic pneumatic control (EPC) flow module in the GC to supply detector gas flow (air).

Detector Design

The OI Analytical Model 5360A XSD consists of two principal components:

- Detector Assembly
- Model 5300 Detector Controller

The Detector Assembly consists of a base assembly (base, adapter, and detector jet), a reactor assembly (including a reactor power cable), and a probe assembly (including a signal probe cable). The Model 5300 Detector Controller houses signal processing, control, and power supply electronics. It is functionally independent of the GC. The detector signal (0–1 V) from the Detector Controller routes to the GC data system either through the GC using an analog interface board (AIB) or through an external analog-to-digital (A/D) box (or similar device).

Operating Principles

The XSD's reactor assembly operates in an oxidative mode to convert halogenated organics to free halogen atoms. Halogen atom adsorption onto the probe surface results in increased thermionic emission from the probe assembly's activated platinum surface. The emission current, composed of free electrons, negative ions, and positive ions, is measured via an electrometer circuit. The voltage corresponding to the measured current exports via a standard BNC connector to the appropriate signal cable. The Detector Controller processes the detector signal, which can be quantified by an external chromatographic integrator or other data handling device. The signal output

from the Detector Controller is internally selectable and ranges from 0 to 1 V DC, 0 to 10 V DC, or 0 to –10 V DC.

The venting option discharges unwanted column effluents before they enter the reactor chamber. Use the venting option for conditioning new columns, venting solvent peaks, and selecting against components eluting within a specific retention window. Activating the valves on the vent valve manifold permits the column effluent to be completely vented away from the detector. Open (energize) both valves at the same time for optimal venting. In the Agilent 6890 GC, connect the venting option through valve five or valve six using an external events cable.

Features

- High halogen selectivity vs. hydrocarbon simplifies analyses and minimizes or eliminates the need for sample preparation.
- High detector sensitivity permits very low-level selective analysis of halogen-containing (Cl, Br, F) compounds.
- The XSD is custom engineered to fit the Agilent 6890 GC, minimizing installing and operating problems.
- The newly-designed detector base heated directly by the GC minimizes baseline rise during temperature programming.
- Easily integrate the XSD into the GC using the external event relay (for solvent venting) and the Agilent analog interface board (AIB).
- A unique base assembly and jet design minimizes peak tailing associated with unswept dead volumes.
- No routine maintenance is required.
- The XSD requires only compressed air for operation.
- The Detector Controller is compatible with the Model 5320 Electrolytic Conductivity Detector (ELCD).

Specifications

General Specifications

Dimensions (Detector Controller)

- 8.3" H x 5.2" W x 12.0" D
  21.0 cm H x 13.2 cm W x 30.5 cm D

Weight (Detector Controller)

- 8.8 lbs (4.0 kg)

Performance Specifications

Dynamic Range

- $>10^5$

Selectivity

- Cl:HC >10^4

Detectivity

- 1 pg Cl/second

Reactor Temperature Range

- 800°, 900°, 1000°, and 1100°C.
- Operator selectable

Gain Settings

- 1, 10, and 100

Signal Output

- 0 to 1 V
- 0 to 10 V via an internal jumper
- 0 to –10 V via an internal jumper

Vent (optional)

- Remotely controlled via the GC's external event relay

Requirements

Gas Supply

CAUTION: Do not use H_2 as the carrier gas. Use only He or N_2.

- 99.999% air as detector gas
- 9.999% He or N_2 as carrier gas

Flow Rate

- 20–40 mL/minute air

Power

- 90–260 VAC, 47–63 Hz, 200 W

Fuse Rating

- 230 V, 1.5A
- 115 V, 3A

Operating Temperature Range

- 5°–40°C

Maximum Relative Humidity

- 80%

Safety Information

The OI Analytical Model 5360A XSD meets the following International Certification when tested in a typical configuration:

- LVD 73/23/eec:1974
- IEC 1010-1: 1990 + A1/EN 61010-1: 1993
- CSA C22.2 No. 1010.1 - 92
- UL 3101, 1st Ed.

The Model 5360A XSD also meets the following Electromagnetic Compliance Certification:

EMC. **Directive 89/336/EEC: 1989**
CISPR 11:1009/EN55011 (1991)
EN 50082-1: 1992
IEC 801-2/EN61000-4-2
IEC 801-3/EN61000-4-3
IEC 801-4/EN61000-4-4

The Model 5360A XSD 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 can impair the instrument's safety protection. Whenever the safety protection of the Model 5360A XSD 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.

Connect the XSD 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.
- 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 do 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 to the instrument that are not OI Analytical original parts. Any unauthorized modifications or substitutions voids the warranty.
- Verify all heated areas have cooled before handling or wear adequate hand protection to prevent burns.

Compressed Gas Cylinder Precautions

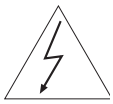
- Store and handle compressed gases in strict accordance with relevant safety codes.
- Fasten all cylinders securely to an immovable structure or permanent wall.
- Store or move cylinders only in a vertical position. Do not move or transport cylinders with the regulators attached.
- Use only approved 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. Handle and store these gases and the cylinders containing them in a manner consistent with OSHA regulations. Maintain adequate ventilation in areas where these materials are used and stored. Avoid prolonged exposure to high concentrations of these gases.
- Oxygen has been identified as an oxidizer. Handle and store these gases and the cylinders containing them in a manner consistent with OSHA regulations. Maintain adequate ventilation in areas where these materials are used and stored. Avoid prolonged exposure to high concentrations of these gases.

Safety Symbols

The following symbols may be located on the instrument:

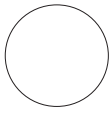
Warning/Caution, see accompanying instruction for more information.

Indicates a hot surface.



Indicates hazardous voltages.

Indicates earth (ground) terminal.



Indicates the OFF position on the power switch.

Indicates the ON position on the power switch.

Chapter 2 Instrument Components

Principal Components

The Detector Assembly includes the base assembly, reactor assembly, and the probe assembly.

- The base assembly consists of a mounting bracket, a jet tube, an adapter fitting, three gas supply lines, a heater cable, an aluminum crunch washer, and a vent valve manifold (optional). Three gas lines are provided:
  - The wall line provides sweep gas for flowing around the outside of the detector jet.
  - The jet line provides column sweep gas through the jet inlet assembly and provides additional gas flow during venting when the venting option is activated.
  - The vent line provides additional sweep gas during normal operation or is used as the exit vent line when the venting option is activated. The gas lines can be supplied from a single gas source.

  A venting option discharges unwanted column effluents before they enter the reactor chamber. The venting option can completely vent a solvent peak, if desired.
- The reactor assembly consists of a reactor case, a reactor core, and a reactor cap. The reactor core is installed within the reactor case, and the reactor cap is installed on top of the reactor cap and sealed with three set screws.
- The probe assembly consists of a sensor assembly, a heat sink, and an electrical connector to the anode and cathode. The assembly ensures reliable operation and is easily replaceable. The probe assembly mounts on top of the reactor cap. Three screws attach the probe assembly to the reactor assembly. A two-pin latching connector and a probe cable that connects to the back of the Detector Controller make the detector bias and signal connections.

The Model 5300 Detector Controller provides reactor temperature control and processes the detector signal using a built-in electrometer circuit.

Detector Assembly, Assembled View

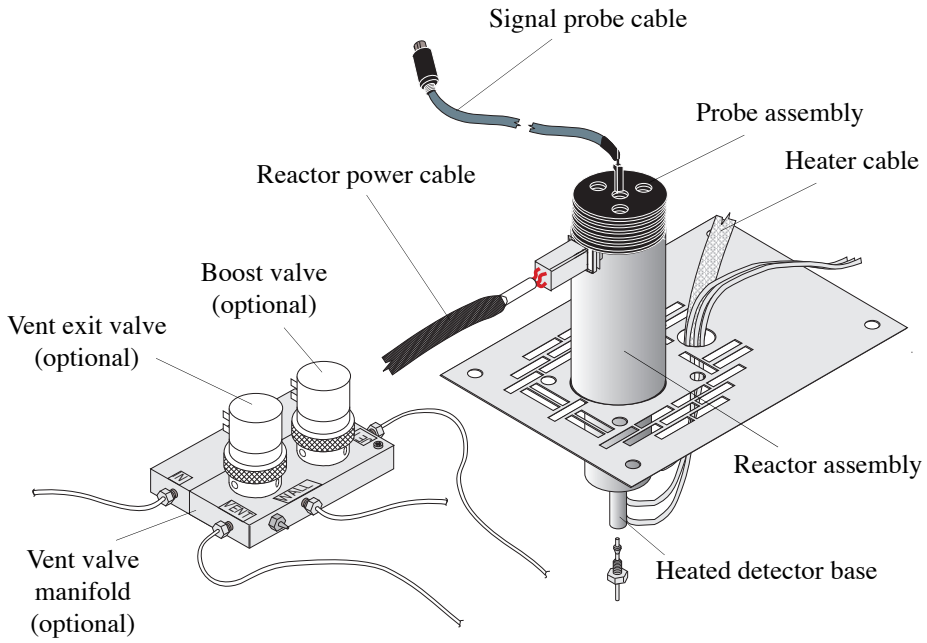


Figure 2.1. Detector Assembly with venting option, assembled view

Boost valve is part of the venting option and provides additional gas supply to the detector base when activated. This additional gas flow actively vents the column effluent out of the detector via the vent exit valve.

Heated detector base is heated by the GC and attaches to the GC using a mounting bracket.

Heater cable consists of a heater and thermocouple through which the GC controls the detector base temperature.

Probe assembly mounts on top of the reactor assembly and contains the signal probe where the halogen reaction and detection occur.

Reactor assembly contains the heat source that enables complete halogen compound oxidation. It contains a removable reactor core.

Reactor power cable supplies power to the reactor assembly. The three-pin latching connector connects to the reactor power supply connector on the back of the Detector Controller. The four-pin connector attaches to the connector on the reactor core.

Signal probe cable connects the probe assembly to the Detector Controller. The end with the two-pin latching connector connects to the probe assembly. The end with the six-pin mini-DIN connector plugs into the corresponding six-pin mini-DIN receptacle (signal in connector) on the back of the Detector Controller. The signal from the signal probe travels through the signal probe cable to the electrometer circuitry in the Detector Controller.

Vent exit valve on the venting option controls the vent port through which the column effluent exits the detector during venting. The GC's external event relay controls both the vent boost valve and the vent exit valve.

Vent valve manifold is the part of the venting option through which the gas supplies are routed to the detector during both normal and venting operation of the XSD. If no venting option is used, replace the vent valve manifold with a cross fitting.

Detector Assembly, Exploded View

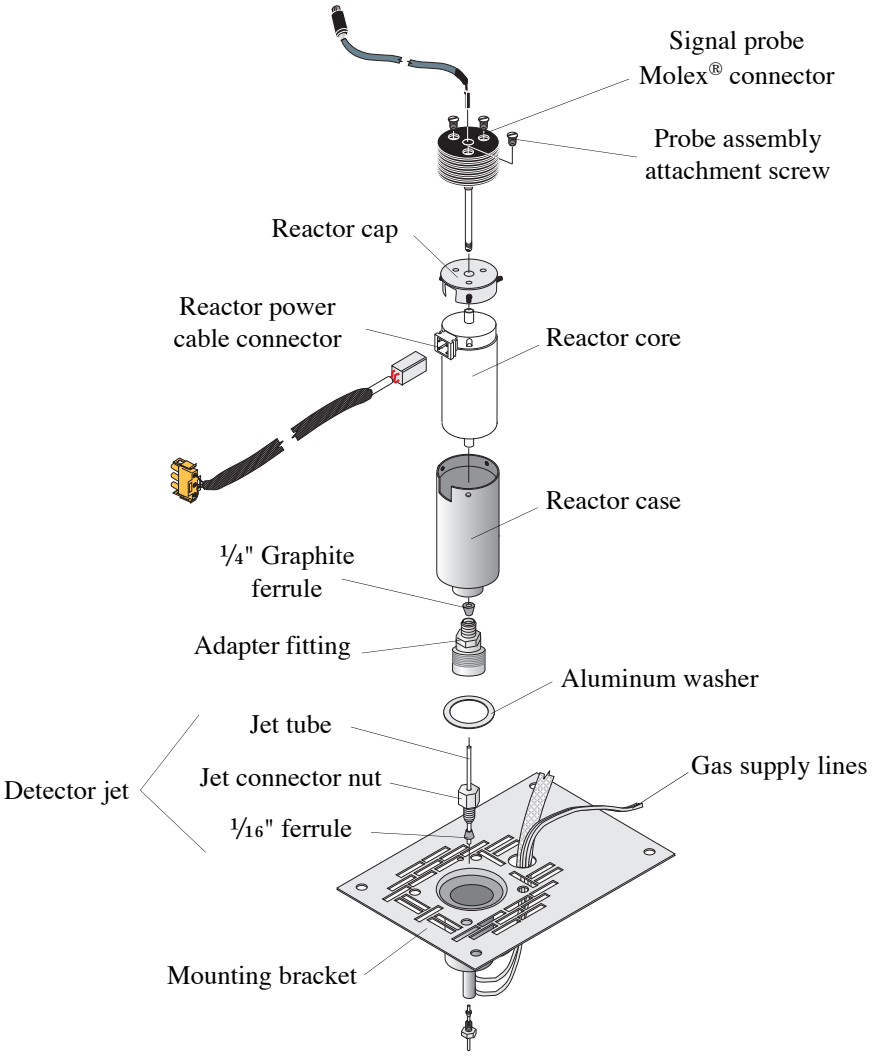


Figure 2.2. Detector Assembly, exploded view

Aluminum washer creates a gas seal between the heated detector base and the adapter fitting.

Detector jet carries the column effluent to the reactor assembly. It consists of a glass-lined jet tube, the jet connector nut, and a 1/16" ferrule.

NOTE: Take care not to bend the jet tube.

NOTE: The 1/16" ferrule mounts in reverse with the tapered end going into the jet connector nut and the flat end pointing toward the end of the jet tube.

NOTE: The XSD uses two different jet tubes: one is used for normal operation (0.8 mm I.D.) and the other is used with the venting option (0.3 mm I.D.).

Gas supply lines connect to either a reliable gas supply regulator or a flow controller. When installed with the venting option, the gas supply lines connect to the vent valve manifold.

Mounting bracket attaches the heated detector base to the top of the GC oven using mounting screws.

Model 5300 Detector Controller, Front View

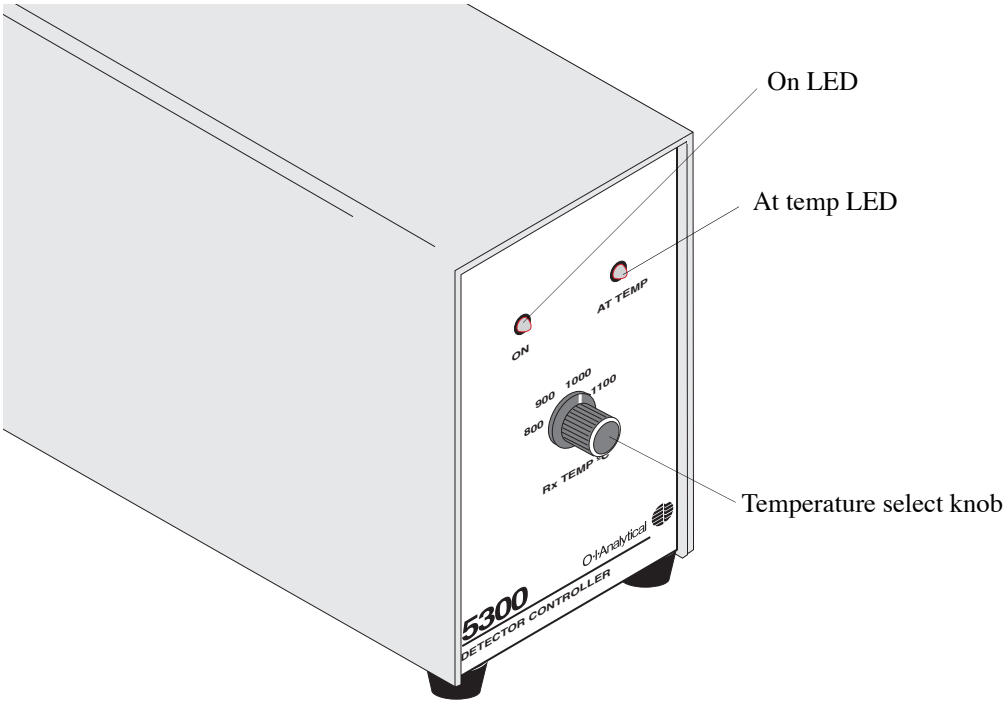


Figure 2.3. Detector Controller, front view

At temp LED lights when the reactor temperature reaches the selected temperature. This LED turns off when the reactor is not at the specified temperature or when the power is off.

On LED indicates the power status.

Temperature select knob (Rx Temp °C) selects the reactor temperature. The standard temperatures are 800°, 900°, 1000°, and 1100°C. The reactor temperature is calibrated at the factory.

Model 5300 Detector Controller, Back View

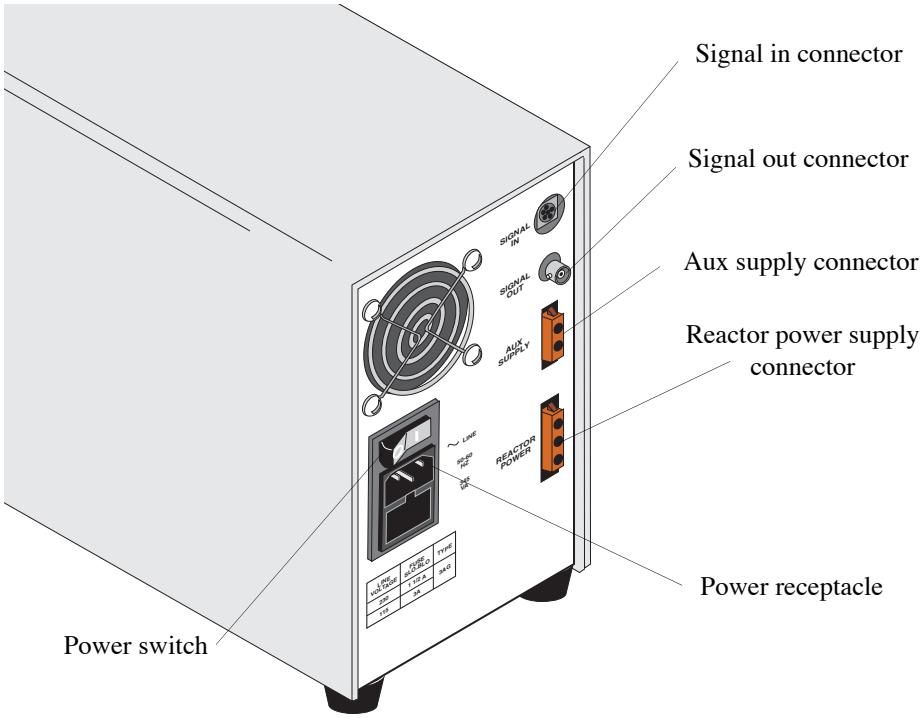


Figure 2.4. Detector Controller, back view

Aux supply connector is not used for XSD operation.

Power receptacle provides electrical power via a power cord and a power source with a protective earth ground.

Power switch (rocker switch) turns the reactor power, bias voltage, and electrometer power on and off. The On LED on the front of the Detector Controller indicates the power status.

Reactor power supply connector (three-pin connector) joins with the reactor power cable to supply power to the reactor assembly.

Signal in connector (six-pin connector) joins with the signal probe cable.

Signal out connector (BNC connector) provides a signal output voltage with a nominal range of 0 to 1 V. The detector output cable connects the Agilent 6890 GC AIB and the Detector Controller. Other cables are available for connecting to other types of data handling devices.

NOTE: Jumper the signal output voltage internally to a nominal range of 0 to 10 V output using the JP7 jumper in the Detector Controller. Switch the polarity from positive to negative through the S2 switch in the Detector Controller.

Chapter 3 Installation

This chapter describes the procedure for installing the Model 5360A XSD. Mount the XSD as a stand-alone detector or in tandem with a photoionization detector (PID). For stand-alone operation, mount the detector on the GC using the heated base. As a tandem detector, mount the XSD directly to the PID. The XSD must be installed by an OI Analytical-qualified technician.

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.

WARNING: Never use H_2 as carrier gas, sweep gas, or makeup gas with the XSD.

Preparing the GC

1. If the GC is not already in place, set it up according to the instructions in the *Agilent 7890 or 6890 Series GC Operator's Manual*.

2. Turn off the power and unplug the power cord from the electrical outlet. Turn off all gas supplies at their sources after the heated zones have cooled.

3. Remove the detector port cover, the electronics compartment top and right side covers, and the back cover(s) from the GC.

4. If present, remove the electrometer board corresponding to the selected detector port. Locate this board in the electronics compartment on the right side of the GC. In the same location, install the appropriate Agilent analog interface board (AIB) (refer to Figure 3.1 and Figure 3.2). For more information, see the *Agilent 7890 or 6890 Series Gas Chromatograph Operator's Manual*.

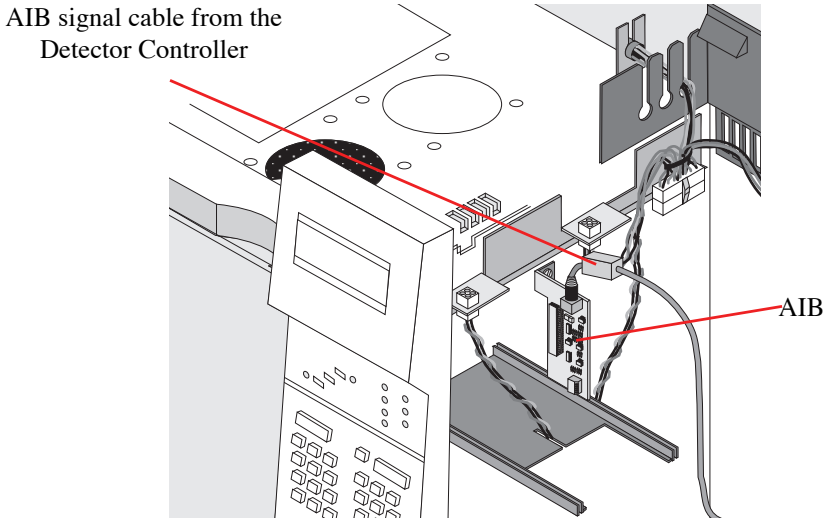


Figure 3.1. Placing the analog input board (AIB) in the Agilent 6890

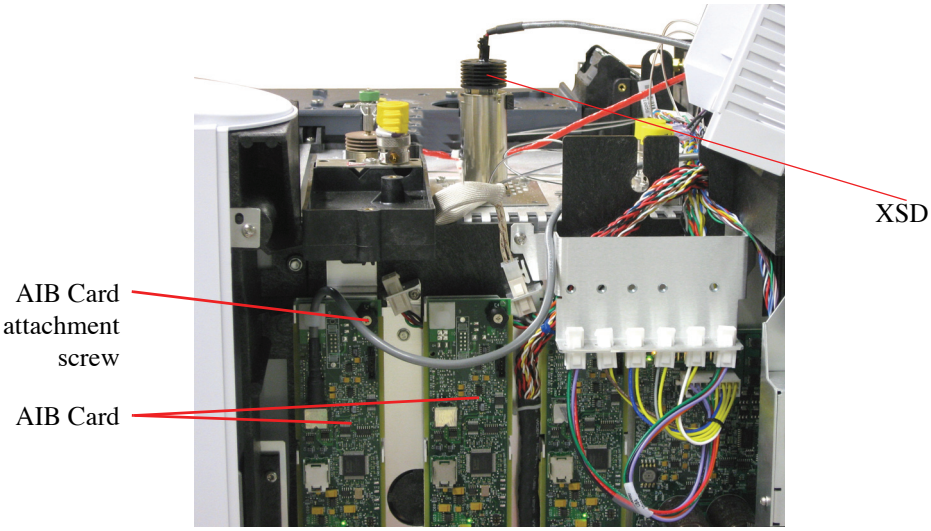


Figure 3.2. Placing the analog input board (AIB) in the Agilent 7890

5. If a detector is already located in the desired detector port for the XSD, allow the existing detector to cool to room temperature. Disconnect all associated gas lines, the GC column, and the electronics. Remove the detector according to the proper procedure described in the detector's operator's manual.

6. Remove the circular, aluminum detector port cover and the circular insulation plug from the desired mounting position in the GC's detector area (refer to Figure 3.3). Set the insulation plug aside for later use.

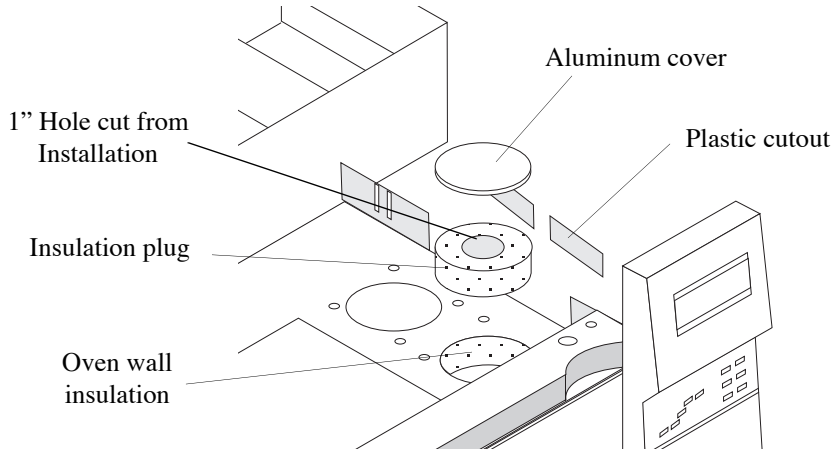


Figure 3.3. Preparing the Agilent GC's detector port

7. Place a paper towel or laboratory tissue on the GC oven's floor.

CAUTION: Do not remove the entire lower level of oven insulation. Leave at least half of the lower insulation in place.

8. From the top of the GC, cut away about half of the lower (hard) oven insulation in the bottom of the detector port. Cut out the diameter shown in Figure 3.4. Remove this insulation to allow the detector to sit properly in the detector port. Do not remove the entire lower level of insulation. Leave at least half of the lower insulation in place.

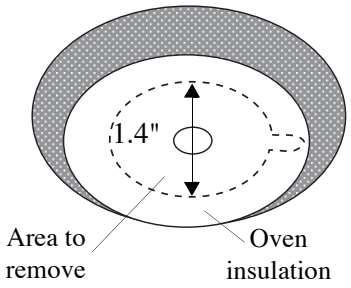


Figure 3.4. Cutting away the oven insulation

9. After cutting away the insulation, carefully remove all the insulation material debris and the paper towel.

10. Remove the plastic cutout to the right of the selected detector port (refer to Figure 3.3).

11. Cut the insulation plug removed from the detector port to fit around the detector base.

Installing the EPC Flow Module

Installing the OIM Pressure Control Manifold on an Agilent 7890

The open interface module (OIM) pressure control manifold is the gas flow control device that connects the main gas source to the XSD. The preferred gas supply option for the XSD is the open interface module (OIM) pressure control manifold (PN 324768), which mounts in the back of the Agilent 7890 GC in one of the EPC ports marked "EPC3" (front detector) or "EPC4" (back detector); refer to Figure 3.6 and Figure 3.6.

NOTE: The OIM pressure control manifold requires well-regulated gas supplies to operate the XSD. Pressure in the 50–70 (maximum 90) psi range is acceptable.

NOTE: All gas line tubing must be chromatography quality to prevent detector noise and instability during operation.

Table 3.1. Gas supplies for the XSD detector configurations

Detector Model	Electronic Pressure Control Configuration	Gas Connections		
		Gas Inlet Port Labels		
		H2	Makeup[1]	Air
5360A XSD	1 x OIM	*Air*[2] *XSD Air* *No venting*	— *Makeup*	Air[2] *XSD Air* *(with venting)*
5390 PID/XSD	1 x OIM	*PID Sweep*	MU[1] *Makeup*	Air[3] *XSD Air*

1 Nitrogen can replace He for makeup gas.

2 When an XSD is installed as a stand-alone detector without the venting option, control air flow through the OIM's H_2 gas inlet and outlet to ensure more accurate air flow rates below 20 mL/minute. This results in actual air flow values being approximately half the set value. However, if the Model 5360A is installed with the venting option, the higher air flow requirement necessitates plumbing the XSD air flow through the regular air inlet and outlet of the OIM. Program flow rates in the 300–500 mL/minute range to provide an optimum flow rate of 20 mL/minute through the XSD. This higher programmed value results in a much lower actual flow to the XSD due to the restrictors in the vent valve manifold. During venting, the actual air flow to the XSD is much higher than during nonventing operation (refer to "Installing the Venting Option" on page 37 in this chapter).

3 When the Model 5390 PID/XSD is installed, the PID sweep gas uses the OIM's H_2 inlet and outlet. Channel the XSD air supply through the OIM Air inlet and outlet. **In addition, use He with this configuration instead of H_2 as the PID sweep gas.**

Install the OIM pressure control manifold into the Agilent 7890 and the gas supply from the OIM to the PID using following procedure.

1. Remove the EPC module bracket by loosening the captive screws and lifting off the brackets (refer to Figure 3.5).

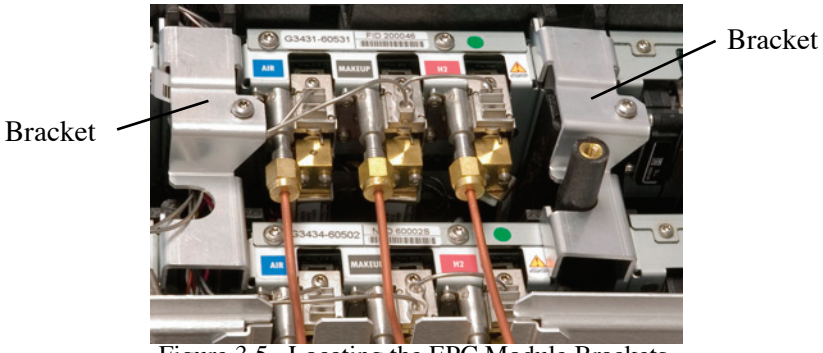


Figure 3.5. Locating the EPC Module Brackets

2. Locate the correct mounting slot for the EPC module. The slot labeled EPC3 is for the front detector position, and the slot labeled EPC4 is for the back detector position (refer to Figure 3.6 and Figure 3.6).

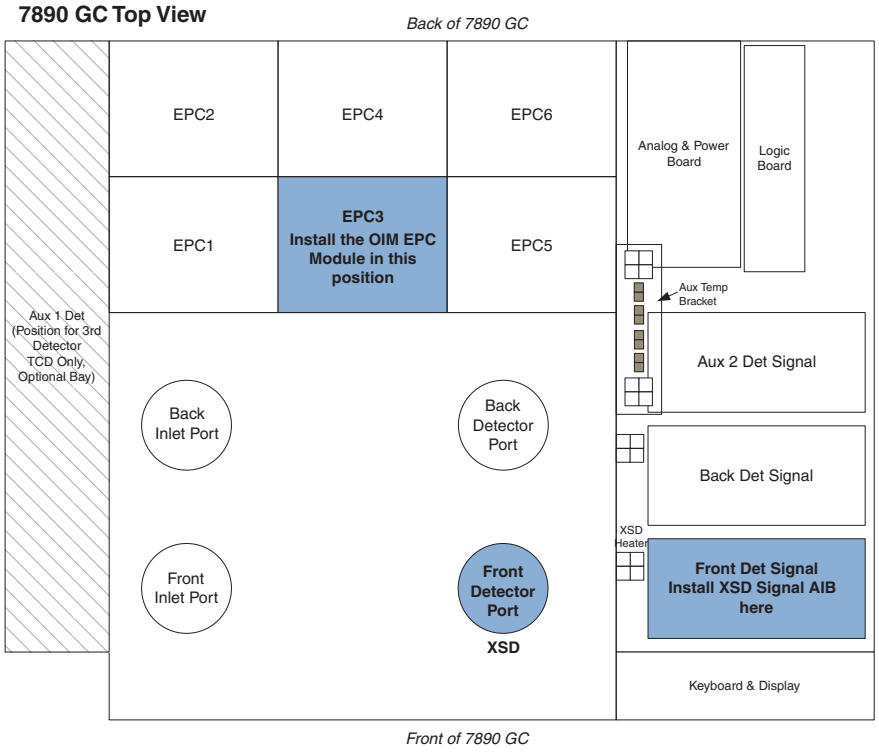


Figure 3.6. Agilent 7890 Top Detector Location - Front Detector Position

NOTE: For other configurations, see Chapter 8, “Appendix” on page 71.

3. Attach the communication cable to the EPC module (refer to Figure 3.7).

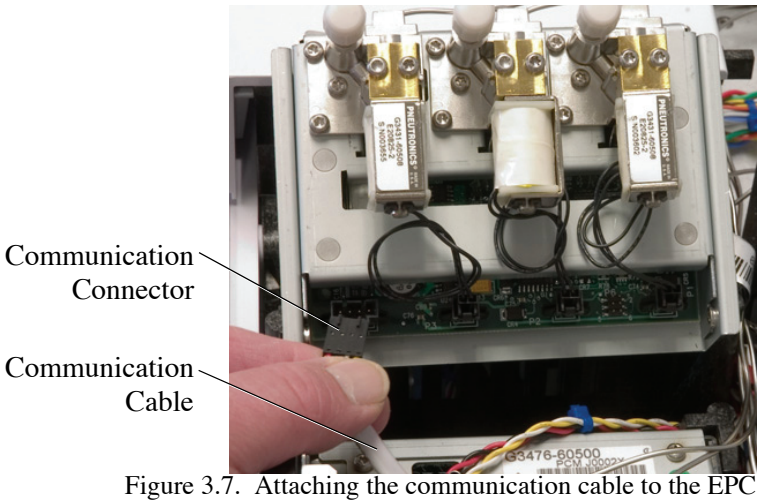


Figure 3.7. Attaching the communication cable to the EPC module

4. Slide the module into the vertical slot tracks. Arrange the cable to prevent it from being pinched by the EPC module (refer to Figure 3.8).

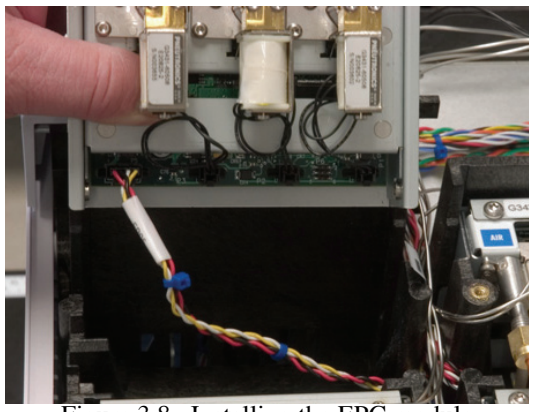


Figure 3.8. Installing the EPC module

Installing the Pneumatic Gas Lines for Standalone XSD on an Agilent 7890 using an OIM Module

1. Remove the protective cap from the appropriate gas inlet using Table 3.1 and Figures 3.10 and 3.11.

2. Connect Air to the appropriate inlet using ⅛" brass nuts and ferrules (refer to Figure 3.10 and 3.10).

3. Remove the appropriate tubing connector blank (refer to Figures 3.9, 3.9, and 3.10) from the EPC module to expose the O-ring.

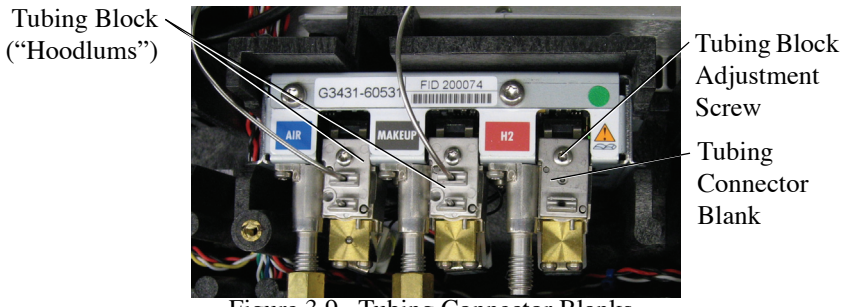


Figure 3.9. Tubing Connector Blanks

4. Attach the tubing block (hoodlum block assembly with integrated 1/16" stainless steel lines) to the appropriate manifold and tighten the screw (refer to Table 3.1).

5. Cut off the ⅛" welded nib on the end of the line.

6. Route the tubing through the channel between the EPC slots to reach the detector area. Keep the tubing away from the EPC module bracket area.

7. Route the 1/16" stainless steel reaction gas (Air line from the OIM module along the tubing guides on the top of the GC and to the detector area.

8. Attach the end of the air delivery line to the cross fitting (PN 319519) using a 1/16" nut and ferrule (included with the detector startup kit).

9. Make sure that the air delivery line is pushed into the cross fitting as far as it can go before tightening the nut. Securely tighten using a 5/16" wrench.

10. Using needle-nosed cutters, remove the appropriate detector cutout from the GC's top back panel.

11. Replace the GC's top back panel.

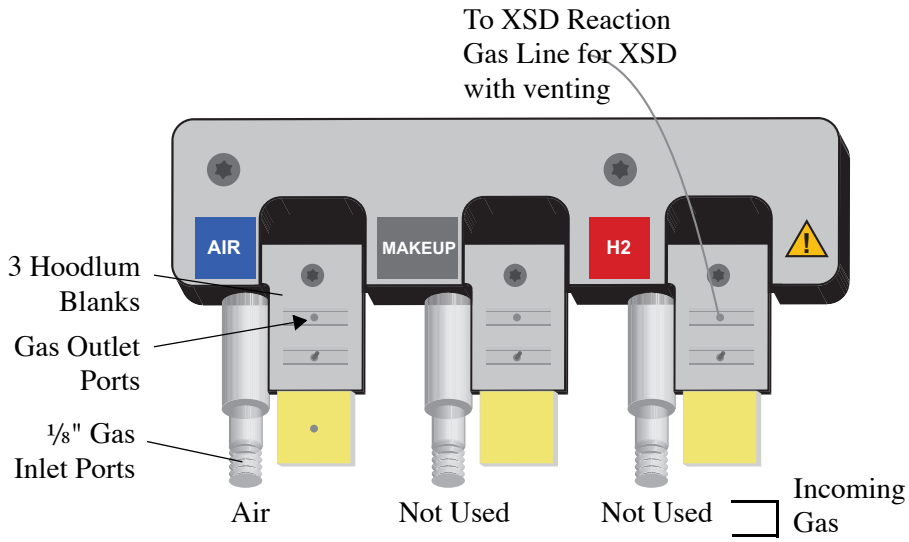


Figure 3.10. OIM Module Plumbing for Standalone XSD with Venting

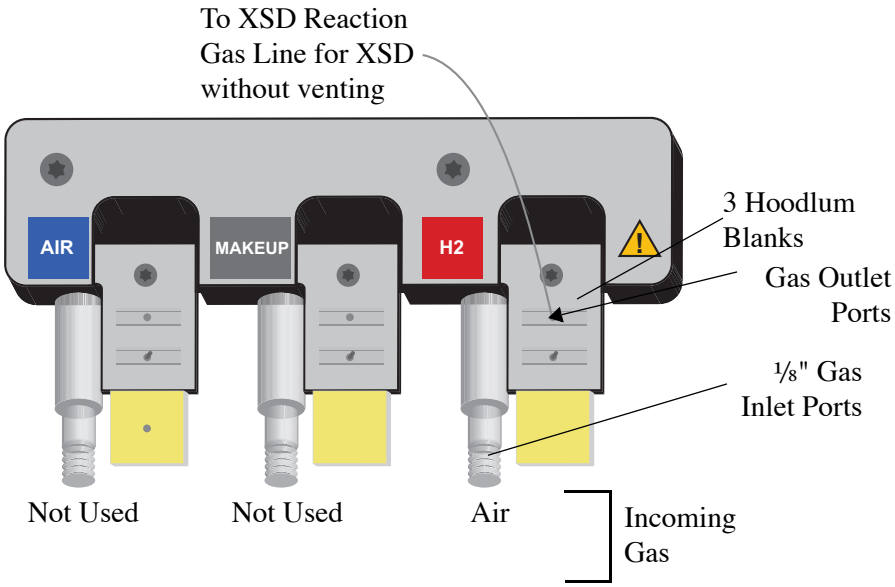


Figure 3.11. OIM Module Plumbing for Standalone XSD without Venting

12. Reattach the EPC module brackets using the previously removed screws.

Standalone XSD using the 7890 Aux EPC

When using the Aux EPC module for control of the XSD, install the XSD into the "back detector" port of the GC. Use the "Back Detector" heater connector. The XSD heater will not work if plugged into the "Front Detector" port when using "Aux EPC". The Aux EPC is not recommended for tandem PID/XSD configuration.

Installing the Aux EPC Module in an Agilent 7890

The auxiliary EPC manifold (Aux EPC) controls the gas flow to the XSD. It mounts in the back of the Agilent 7890 in either port 4, 5, or 6. Use the following procedure to attach the XSD's gas flow controls to the auxiliary pressure control manifold.

NOTE: The auxiliary EPC manifold requires a well-regulated supply of Air and air. Pressures in the 50–70 (maximum 90) psi range are acceptable.

CAUTION: The maximum pressure into the EPC must not exceed 90 psi.

Preparing the GC

1. Shut off all gas supply lines used for the XSD.

NOTE: Use chromatography-quality gases of 99.999% purity or better.

NOTE: Use chromatography-quality gas line tubing to minimize background noise during operation.

2. Turn off and unplug the GC.

3. Remove the gas flow controls cover by pressing the black clips on the sides of the cover.

4. Remove the detector cover by raising the cover vertically and then firmly lifting up on the right side of the cover to free the lid from the hinge pin.

5. Slide the hinge pin out of the hole on the left side hinge and set the cover aside.

6. Remove the EPC module bracket at the empty slot location by loosening the captive screw and lifting off the bracket.

Assembling the Aux EPC

1. Remove the AUX1 tubing connector blank (refer to Figure 3.12) from the Aux EPC module to expose the O-ring and restrictor (leave the other two blanks in place).

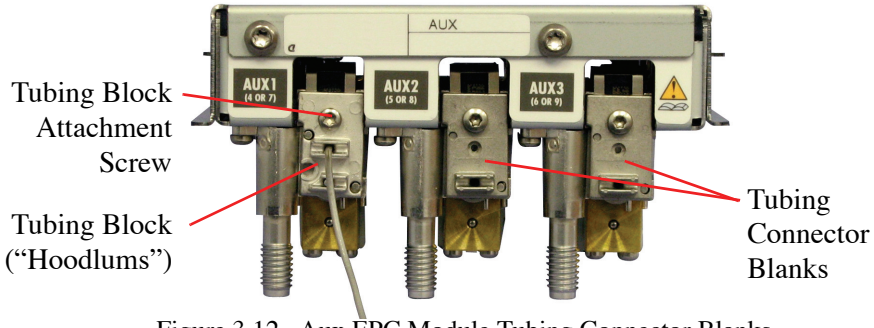


Figure 3.12. Aux EPC Module Tubing Connector Blanks

2. Change to the frit labelled "30 ±1.5 sccm H_2 at 15 psi" for each of the Aux gases to be utilized. The Aux EPC module is shipped with brown color-coded, low-resistance (high flow) frits.

3. Attach the tubing blocks (hoodlum block assembly with integrated 1/16" stainless steel lines) to each of the manifolds to be utilized and tighten each screw (refer to Figure 3.12 and Figure 3.13 or Figure 3.12).

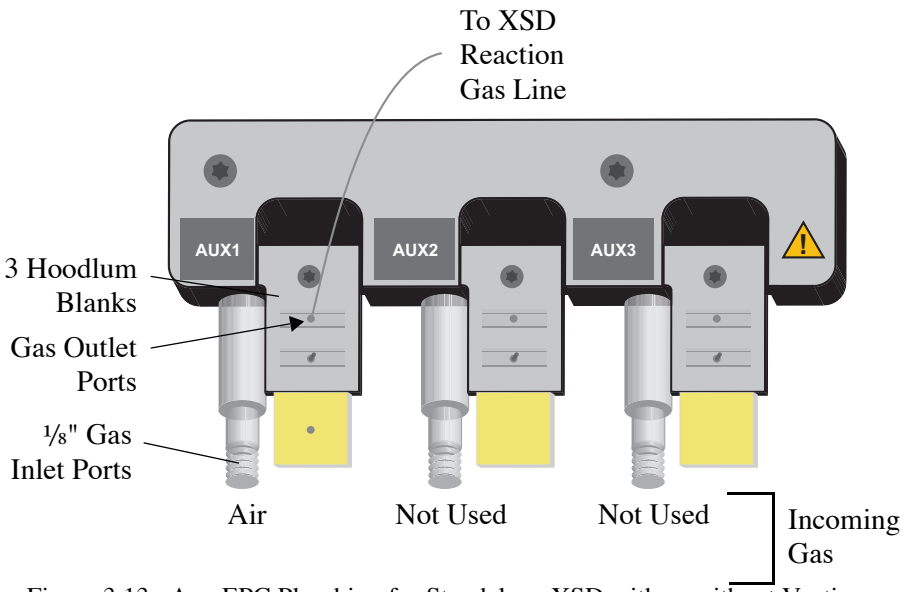


Figure 3.13. Aux EPC Plumbing for Standalone XSD with or without Venting

Installing the Aux EPC

1. Determine the slot position for your Aux EPC module in the gas flow control area. The slots are numbered 1–6 (refer to Figure 3.14); the Aux EPC can be installed in slots 4, 5, or 6 (installation in slot 6 is recommended).

Slot 5	Slot 3	Slot 1	Split vent traps and valves
Slot 6	Slot 4	Slot 2	

Back of 7890 GC (gas flow control compartment)

Figure 3.14. Slot Identification

2. Record the inlet channel identification numbers associated with your slot position. These channel numbers are used during the hardware configuration. (refer to Figure 3.15)

Aux EPC 4, 5, 6	Slot 3	Slot 1	Split vent traps and valves
Aux EPC 1, 2, 3	Aux EPC 7, 8, 9	Slot 2	

Back of 7890 GC

Figure 3.15. Aux EPC inlet channel identification numbers

3. Connect the communication cable for your EPC slot location to the communication connector on the Aux EPC module.

4. Slide the Aux EPC module into the vertical slot tracks. Arrange the cable to prevent it from being pinched by the Aux EPC.

5. Route the tubing through the back EPC cover panel to exit the GC. Keep the tubing away from the EPC module bracket area.

6. Reattach the EPC module bracket by tightening the captive screw.

7. Remove the protective caps from the Aux EPC module source gas connections.

Installing the OIM Gas Flow Module 6890

The pressure control manifold is the gas flow control device between the main gas source and the XSD. The preferred gas supply option for the XSD is the open interface module (OIM) gas flow module (PN 285049), which is mounted in the back of the Agilent 6890 GC in one of the pneumatics carrier ports marked “Front Detector” or “Back Detector”. OI Analytical programs the OIM gas flow module for the desired detector configuration before shipping. Refer to Table 3.2 for the gas inlet and outlet configurations that apply when the OIM gas flow module is used. Alternatively, supply the gas through an AUX pressure control manifold (PN 275974), which mounts in the pneumatics carrier port marked “AUX”. See “Installing the Auxiliary Pressure Control Manifold on 6890” on page 27 in this chapter.

CAUTION: Do not use H_2 as the carrier gas.

NOTE: Both the OIM gas flow module and AUX pressure control manifold require an air supply with well-regulated pressure to operate the XSD. Pressure in the 50–70 (maximum 90) psi range is acceptable. Use chromatography-quality tubing for all gas lines to avoid detector noise or unstable operation.

Table 3.2. Gas supply for the Model 5360A XSD and the Model 5390 PID/XSD

Detector Model	Pressure Control Manifold	Gas Inlet and Outlet Port *OIM Detector Gas Labels*		
		H_2	**Makeup**	**Air**
5360A XSD	1 x OIM or AUX	Air *XSD Air* (no venting)	—	Air *XSD Air* (with venting)
5390 PID/XSD	1 x OIM or AUX	He *PID Sweep*	MU *Makeup*	Air *XSD Air*

- Makeup (MU) gas can be either He or N_2.
- When an XSD is installed as a stand-alone detector without the venting option, control air flow through the OIM's H_2 gas inlet and outlet to ensure more accurate air flow rates below 20 mL/minute. This results in actual air flow values being approximately half the set value. However, if the Model 5360A is installed with the venting option, the higher air flow requirement necessitates plumbing the XSD air flow through the regular air inlet and outlet of the OIM. Program flow rates in the 300–500 mL/minute range to provide an optimum flow rate of 20 mL/minute through the XSD. This higher programmed value results in a much lower actual flow to the XSD due to the restrictors in the vent valve manifold. During venting, the actual air flow to the XSD is much higher than during nonventing operation (refer to "Installing the Venting Option" on page 37 in this chapter).
- When the Model 5390 PID/XSD is installed, the PID sweep gas uses the OIM's H_2 inlet and outlet. Channel the XSD air supply through the OIM Air inlet and outlet. **In addition, use He with this configuration instead of H_2 as the PID sweep gas.**

Use the following procedure to install the OIM gas flow module into the Agilent 6890 GC and the gas supply from the OIM gas flow module to the XSD. The required parts are included in the supplied detector gas kit (PN 251892).

1. Shut off all gas supply lines used for the XSD pneumatic system. Remove the top back panel on the GC.

2. Remove the Agilent gas outlet block attached to the front upper right corner of the OIM gas flow module by unscrewing the Torx T-20 outlet block attachment screw (PN 185561) (refer to Figure 3.16). Leave the three O-rings in place

.

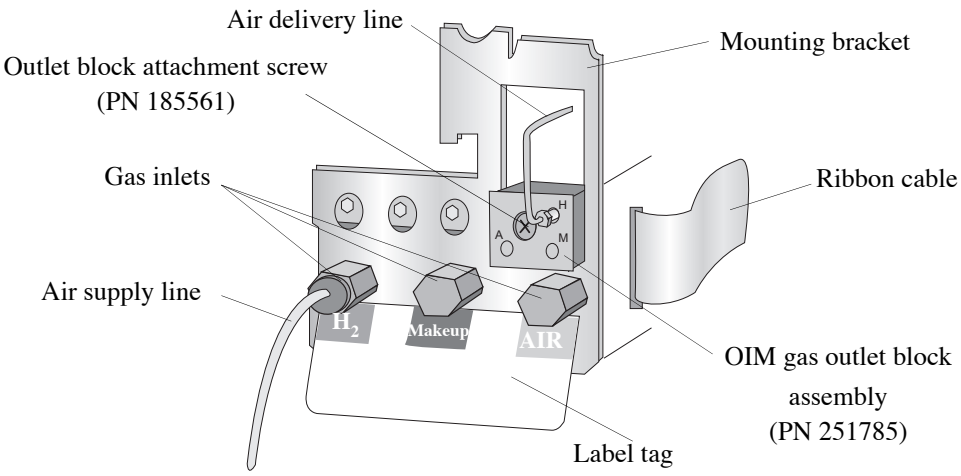


Figure 3.16. OIM gas flow module (nonventing configuration)

3. With the three O-rings in place, attach the OIM gas outlet block assembly (PN 251785) to the OIM gas flow module using the outlet block attachment screw. Make sure that the OIM gas outlet block is flush with the OIM gas flow module.

4. Remove the protective caps from the gas inlets.

5. Slip the label tag through the bottom slot of the gas flow module mounting bracket. Align the mounting bracket over the OIM gas flow module.

6. Place the 7/16" hex nuts provided on the gas inlets. Do not tighten the hex nuts.

7. Tighten the 7/16" hex nuts on the gas inlets and secure the mounting bracket to the OIM gas flow module. Replace the protective caps on the gas inlets.

8. Route the ribbon cable behind the OIM gas flow module.

9. Slide the OIM gas flow module into the appropriate “Front Detector” or “Back Detector” slot on the back of the GC until the bracket seats flush with the carrier rails.

10. Connect the ribbon cable to the appropriate connector on the electronic board. Push the ribbon cable until it is firmly in place. Lock the connector by moving the tabs to the center of the connector until they click into place. Make sure that the ribbon cable is away from the valves and is not pressed against the OIM gas flow module.

11. If not using a venting option, remove the protective cap from the gas inlet marked “H_2” on the front of the gas flow module. Attach the air supply line to this inlet using a 1/8" brass Swagelok® nut and brass ferrule (refer to Figure 3.16).

12. If using a venting option, remove the protective cap from the gas inlet marked “Air” on the gas flow module front. Attach the air supply line to this inlet using a ⅛" brass Swagelok nut and brass ferrule (refer to Figure 3.17).

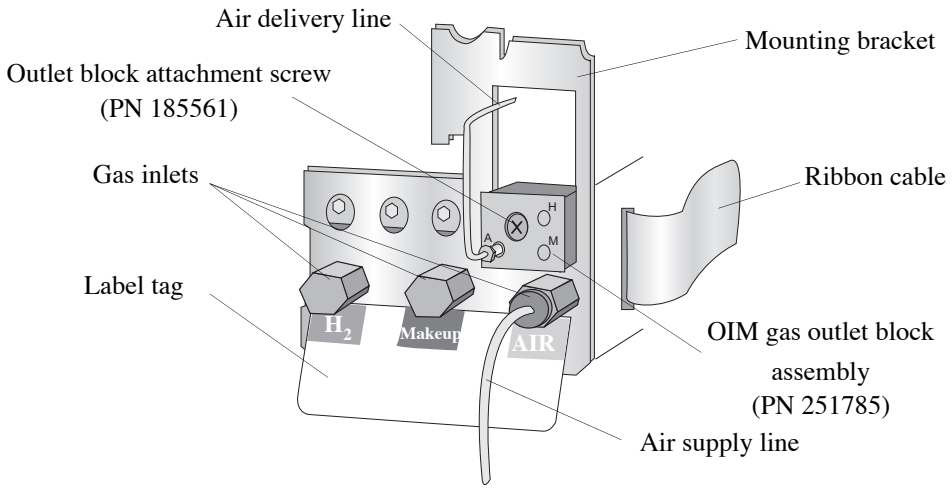


Figure 3.17. OIM gas flow module (venting option configuration)

13. Make sure that the air supply line is pushed into the relevant connector as far as it can go before tightening the nut onto the connector. Securely tighten the ⅛" nut using a 7⁄16" wrench.

14. If the venting option is not used, attach the supplied 1⁄16" stainless steel air delivery line (PN 193409) to the gas outlet marked as “H” on the OIM outlet block using a 1⁄16" nut (PN 223057) and ferrule (PN 216366) (refer to Figure 3.16). This line provides oxidant gas flow in the 20–40 mL/minute flow range to the XSD.

NOTE: The tapered end of the reverse ferrule (PN 216366) points into the 1⁄16" nut (PN 223057)—not into the OIM module.

NOTE: Due to the XSD’s low flow requirement, the air supply routes through the H_2 gas flow line instead of the air supply line to provide more accurate flow control.

15. If using the venting option, attach the supplied 1⁄16" stainless steel air delivery line (PN 193409) to the gas outlet marked as “A” on the OIM outlet block using a 1⁄16" nut (PN 223057) and ferrule (PN 216366) (refer to Figure 3.17). This line provides oxidant gas flow in the 20–40 mL/minute flow range to the XSD.

NOTE: If the XSD is installed with the venting option (see “Installing the Venting Option” on page 37 of this chapter), program flow rates in the 300–500 mL/minute range to provide an optimum flow rate of 20 mL/minute through the XSD. Make sure that the supply gas line is routed through the air channel (connection) of the EPC module.

16. Make sure that the air delivery line is pushed into the gas outlet block as far as it can go before tightening the nut into the gas outlet block. Securely tighten the nut using a 5⁄16" wrench.

17. Plug the remaining two gas outlets with the 1/16" nuts (PN 233057) and no-hole ferrules (PN 197079) provided. If using the venting option, go to step 20.

18. Attach the opposite end of the air delivery line to the cross fitting (PN 319519) using a 1/16" nut and ferrule (included with the detector startup kit).

19. Make sure that the air delivery line is pushed into the cross fitting as far as it can go before tightening the nut. Securely tighten using a 5/16" wrench.

20. Using needle-nosed cutters, remove the appropriate detector cutout from the GC's top back panel.

21. Replace the GC's top back panel.

Installing the Auxiliary Pressure Control Manifold on 6890

The pressure control manifold is the gas flow control device between the main gas source and the XSD. Supply gas through an AUX pressure control manifold (PN 275974), which mounts in the pneumatics carrier port marked "AUX." Refer to the *Agilent 6890GC Auxiliary Pressure Manifold Installation* for further information.

NOTE: Both the OIM gas flow module and AUX pressure control manifold require an air supply with well-regulated pressure to operate the XSD. Pressure in the 50–70 (maximum 90) psi range is acceptable. Use chromatography-quality tubing for all gas lines to avoid detector noise or unstable operation.

Use the following procedure to install the AUX pressure control manifold into the Agilent 6890 GC and the gas supply from the AUX pressure control manifold to the XSD.

1. Shut off all gas supply lines used by the XSD pneumatic system. Unplug the GC and remove the top back panel.

2. Remove the Agilent gas outlet block attached to the front upper right corner of the AUX pressure control manifold by unscrewing the Torx T-20 outlet block attachment screw (PN 185561) (refer to Figure 3.18).

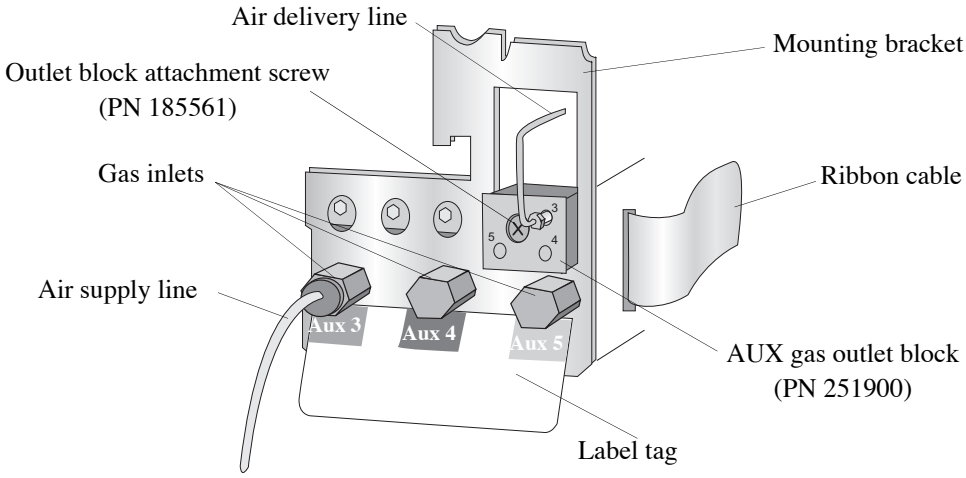


Figure 3.18. AUX pressure control manifold

3. If using the venting option, remove the O-ring and 100–mL restrictor (red dot, Agilent PN 19231-60770) from port 3. Replace the standard restrictor with a 1,000-mL/minute restrictor (PN 295311). Place the removed O-ring over the new restrictor. If the venting option is not used, do **not** replace 100–mL restrictor.

4. With the three restrictors and O-rings in place, attach the AUX gas outlet block (PN 251900) to the AUX pressure control manifold using the outlet block attachment screw. Make sure that the AUX gas outlet block is flush with the AUX pressure control manifold.

5. Remove the protective caps from the gas inlets.

6. Slip the label tag through the bottom slot of the gas flow module mounting bracket. Align the mounting bracket over the AUX pressure control manifold.

7. Place the 7/16" hex nuts provided on the gas inlets. Do not tighten the hex nuts.

8. Tighten the 7/16" hex nuts on the gas inlets and secure the mounting bracket to the AUX pressure control manifold. Replace the protective caps on the gas inlets.

9. Route the ribbon cable behind the AUX pressure control manifold.

10. Slide the AUX pressure control manifold into the appropriate “Front Detector,” “Back Detector,” or “AUX” slot on the back of the GC until the bracket seats flush with the carrier rails.

11. Connect the ribbon cable to the appropriate connector on the electronic board. Push in the ribbon cable until it is firmly in place. Lock the connector by moving the tabs to the center of the connector until they click into place. Make sure that the ribbon cable is away from the valves and is not pressed against the AUX pressure control manifold.

12. Remove the protective cap from the gas inlet marked “AUX 3” on the front of the AUX pressure control manifold, and attach the air supply line to this inlet using a ⅛" brass Swagelok nut and brass ferrule (not supplied).

13. Make sure that the air supply line is pushed into the relevant connector as far as it can go before tightening the nut onto the connector. Securely tighten the ⅛" nut using a 7⁄16" wrench.

14. Attach the supplied 1⁄16" stainless steel air delivery line (PN 193409) to the gas outlet marked as “3” on the AUX pressure control manifold using a 1⁄16" nut (PN 223057) and ferrule (PN 216366). This line provides oxidant gas flow in the 20–40 mL/minute flow range to the XSD.

NOTE: The tapered end of the reverse ferrule (PN 216366) points into the 1⁄16" nut (PN 223057)—not into the AUX EPC block.

NOTE: Due to the XSD’s low flow requirement, route the air supply through the H_2 gas flow line instead of the air supply line to provide more accurate flow control.

15. Make sure that the air delivery line is pushed into the gas outlet block as far as it can go before tightening the nut into the gas outlet block. Securely tighten the nut using a 5⁄16" wrench.

16. Plug the remaining two gas outlets with the 1⁄16" nuts (PN 233057) and no-hole ferrules (PN 197079) provided.

17. Using needle-nosed cutters, remove the appropriate detector cutout from the GC’s top back panel.

18. Replace the GC’s top back panel.

Installing the Model 5300 Detector Controller

1. Set the Detector Controller on the right side of the GC.

2. Verify the power switch on the back of the Detector Controller is in the Off position.

3. If an Agilent 6890 or 7890 AIB has been installed to route the detector signal through the GC, connect the AIB signal cable (PN 274852) from the GC (Figure 3.2) to the signal out connector on the back of the Detector Controller (Figure 2.4).

CAUTION: For 220 V operation, verify the proper fuse is installed in the Detector Controller before connecting to a power source.

4. If an Agilent integrator or an A/D interface is used, connect the BNC connector of the Agilent integrator cable (PN 229633) to the signal out connector on the back of the Detector Controller (refer to Figure 2.4). Connect the other end of the integrator cable to the Agilent integrator or to the A/D interface. Other signal cables are available for other data handling systems. Contact OI Analytical for information on these cables.

5. If not using an AIB or an Agilent integrator, connect the BNC connector of the spade lugs cable (PN 215962) to the signal out connector on the back of the Detector Controller (refer to Figure 2.4).

6. Connect the power cord (PN 116038 for 110 V power supply) to the power receptacle on the back of the Detector Controller. Connect the other end of the power cord to a 115 (±10) VAC power source.

Installing the Model 5360A XSD

Installing the Model 5360A XSD on an Agilent 6890 or 7890 GC

Use the following procedure for installing this GC/detector configuration:

1. Carefully remove the Model 5360A XSD assembly from its packaging.

2. Before installing the XSD onto the GC, make sure that a protector plug is inserted into the GC column attachment at the bottom of the detector base. If not, insert a no-hole ferrule (PN 197079) into the GC column nut (PN 223057), with the tapered end of the ferrule facing into the nut, and screw the nut into the bottom of the detector base to prevent debris from entering the detector base during installation.

CAUTION: Do not kink the gas supply lines extending from the bottom of the detector. Do not drop any loose pieces of insulation material into the detector base.

3. Place the cutout insulation plug (removed in step 6, “Preparing the GC” on page 15 in this chapter) around the detector base below the mounting bracket so that none of the detector base remains exposed.

4. Carefully press the detector base and surrounding insulation into the previously-cut hole in the selected detector port. Ensure the detector base protrudes into the GC oven cavity and that the detector mounting bracket is flush with the metal cover in the detector area. The entire detector base **must** be completely insulated. Fill in any spaces with insulation.

5. Align the mounting bracket with the screw holes corresponding to the selected detector port and secure the mounting bracket using the mounting screws (PN 252585).

6. Connect the heater cable connector to the corresponding four-pin nylon connector located to the right of the detector port above the motherboard in the Agilent 6890 (Figure 3.19) or 7890 GC (Figure 3.20). The front detector port corresponds to the front connector and the back detector port corresponds to the back connector.

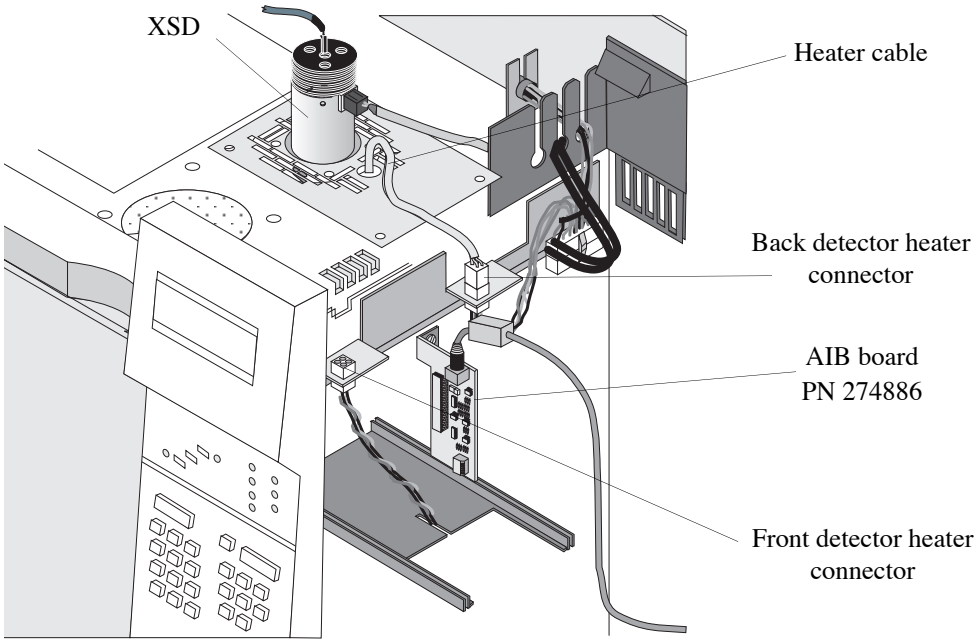


Figure 3.19. Placing the Model 5360A XSD on the Agilent 6890

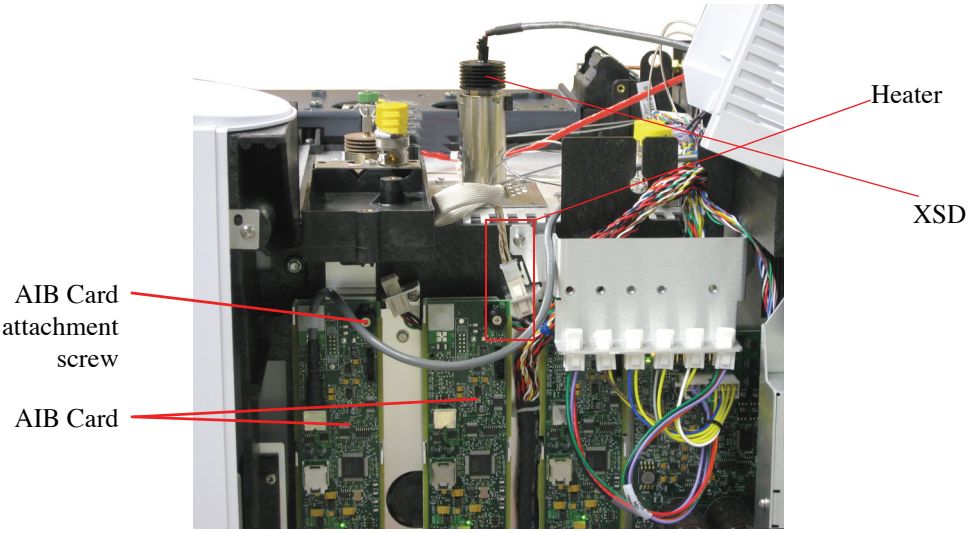


Figure 3.20. Placing XSD on 7890

7. If using the venting option, go to “Installing the Venting Option” on page 37 in this chapter.

8. Using 1⁄16" nuts and ferrules (PN 112433), attach the three gas supply lines from the detector base to the ends of the cross fitting (PN 319519) that is attached to the 1⁄16" stainless steel air supply line connected to the gas flow module (see “Installing the OIM Gas Flow Module 6890” on page 23 or “Installing the OIM Pressure Control Manifold on an Agilent 7890” on page 16 of this chapter).

9. If the adapter fitting (PN 315010) is not installed in the detector base, place an aluminum washer (PN 319510) in the bottom of the detector base, making sure not to scratch the bottom surface of the detector base. Attach the adapter fitting to the detector base using the ½" nut driver tool (PN 319509C) supplied with the detector startup kit.

10. Connect the flow measurement adapter (PN 319631) to the adapter fitting in the detector base. Adjust the gas flow to provide 20–40 mL/minute of total (wall and jet) air flow from the detector base. Remove the flow measurement adapter. Measure the flow out of the jet tube. The flow from the jet tube should be approximately half of the total air flow. The split of the flow is not critical unless <¼ of the total flow is coming through the jet tube.

CAUTION: Lightly fingertighten the reactor base assembly to the detector base. Excessive force damages the reactor core.

11. Carefully screw the reactor assembly (PN 273615) into the detector base. Be careful not to break the reactor core. Fingertighten snugly to seal the reactor assembly to the base. The ¼" graphite ferrule (PN 273623) is installed onto the tested XSD assembly at the factory.

12. If not already attached, attach the probe assembly (PN 251744) by inserting it into the reactor cap (refer to Figure 3.21). Tighten the three mounting screws (PN 132019).

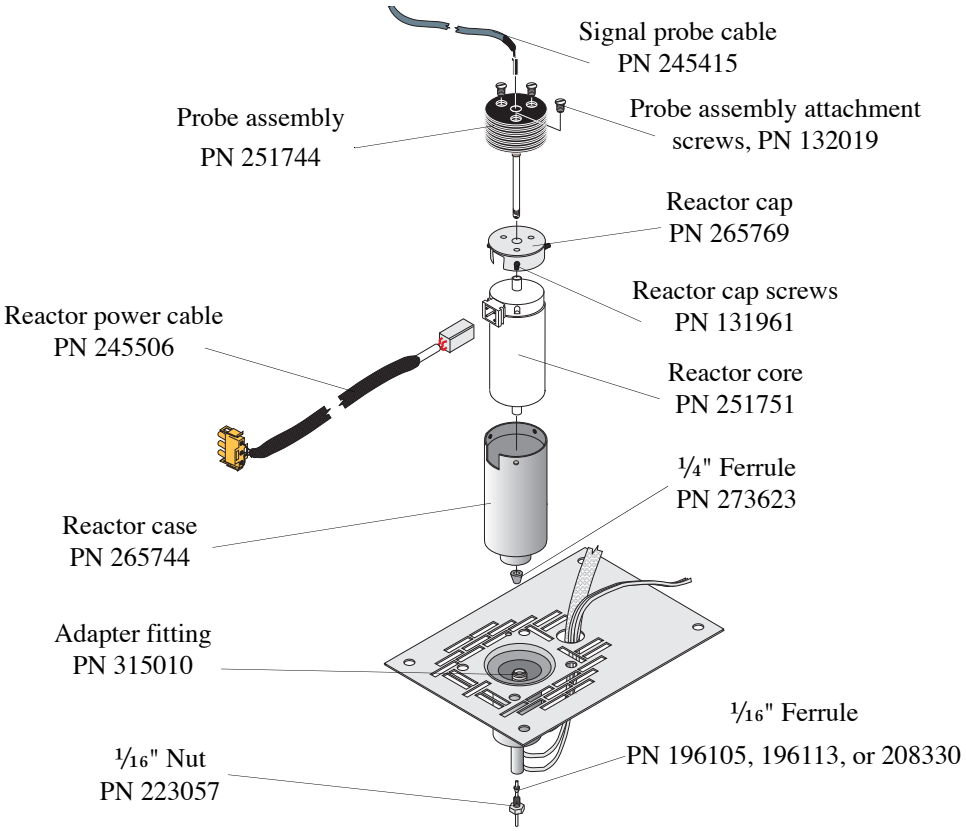


Figure 3.21. Model 5360 Heated XSD Detector Assembly

13. Attach the signal probe cable to the Molex connector on the top of the probe assembly. Route the other end of the signal probe cable to the signal in connector on the back of the Detector Controller (refer to Figure 2.4).

14. Connect the four-pin connector of the reactor power cable to the reactor. Connect the other end of the reactor power cable (PN 245506) to the three-pin reactor power supply connector on the back of the Detector Controller (refer to Figure 2.4). The power cable connector is keyed to ensure proper orientation when connecting to the reactor assembly. Both possible orientations on the connector work properly.

15. Cut an opening in the GC detector cover to allow the XSD reactor assembly and probe to protrude through it.

Configure the Agilent 7890 GC

NOTE: Configuration is not required for an Agilent 6890 GC.

1. Power on the GC.
2. Unlock the keyboard by pressing **Options** and selecting **Keyboard & Display**.
3. Scroll to **Hard Configuration lock**.
4. Press **Off/No**.

Configure the Standalone XSD Configuration with an OIM Module

1. Press **Config** and then either **Front Det** or **Back Det**, depending upon your configuration.
2. Press **Mode/Type**.
3. Scroll to the selection **CPDET AIB, with Htr, EPC*** and press **Enter**.
4. Soft power-cycle the GC as follows:
   a. Press **Options**.
   b. Scroll to **Communications** and press **Enter**.
   c. Scroll to **Reboot GC**.
   d. Select **On/Yes** twice.
5. Once the GC has restarted, press **Config** and then **Front Det** or **Back Det**, depending upon your configuration.
6. Turn off the GC.

Configure the Standalone XSD Configuration with an Aux EPC Module

Configure the Aux EPC Module

1. Press **Config** and then **Aux EPC #**.
2. Scroll to the appropriate Aux EPC position and press **Enter**. Refer to Figure 8.3 and Figure 8.6.

   For example, if the Aux EPC module is installed in the EPC6 position, select **Pressure Aux 1,2,3**.
3. Press **Mode/Type**.
4. Press **Enter** to select the appropriate EPC module.

For the previous example, **Install EPC6** would be selected.

5. Soft power-cycle the GC as follows:

   a. Press **Options**.

   b. Scroll to **Communications** and press **Enter**.

   c. Scroll to **Reboot GC**.

   d. Select **On/Yes** twice.

6. Once the GC has restarted, press **Config** and then **Aux EPC #**.

7. Scroll to the appropriate Aux EPC position and press **Enter**.

   For example, if the Aux EPC module is installed in the EPC6 position, select **Pressure Aux 1,2,3**. The Aux EPC channels are labeled 1, 2, and 3; Channel 1 is Hydrogen for the reaction gas, and Channels 2 and 3 are not used.

8. In this case, scroll to **Chan 1 Gas Type**.

9. Press **Mode/Type**.

10. Scroll to **Air** and press **Enter**.

Configure the AIB Board(s)

1. Determine where the AIB board(s) was installed using Figure 8.6.

2. Press **Config** and then the appropriate detector button (either **Front Det**, **Back Det**, or **Aux Det #**).

   For **Aux Det #**, scroll to select **Aux Det 2**.

3. Press **Mode/Type**.

4. For **Front Det** or **Back Det**, select **CPDET AIB, No Htr, No EPC**.

   For **Aux Det #**, press **Enter** to select **Install Detector (AIB)**.

5. Cycle the GC power.

Check Configurations

1. Press the **Status** key to determine if the GC is reporting any errors.

2. Following the previous configuration example, press **Aux EPC #** and **1** to view the Air channel. Confirm that the Aux pressure reading displays for this channel.

3. Note where the AIB card(s) is installed. Press the key corresponding to the appropriate AIB signal.

   For example, if the AIB card is installed in the Front Detector signal board position, press the **Front Det** key.

4. Verify the AIB signal displays.

Other Detector Configurations

For other detector configurations, refer to "Installing the Model 5360 XSD onto the PID" on page 39 and Chapter 8, "Location of Detectors, Modules, and Heaters for an Agilent 7890" on page 71.

* Other detector configuration selections are listed in Figure 8.1 through Figure 8.5 starting on page 71.

Installing the GC Column

1. Using Table 3.3, select the appropriate ferrule to install the capillary column into the detector. Slide a 1/16" nut (PN 223057) and the selected ferrule over the end of the column.

Table 3.3. Column diameter and corresponding ferrule size

Column I.D.	Ferrule I.D.	Part Number
0.53 mm	0.8 mm	196105
0.32 mm	0.5 mm	196113
<0.32 mm	0.4 mm	208330

CAUTION: Do not use H_2 as the carrier gas. Only use He or N_2.

2. Trim the top of the column using a column scribe. Ensure the column top is clean and has been squarely cut. If necessary, wipe the top of the column with a clean methanol-moistened, lint-free laboratory tissue to remove any dust or finger smudges.

3. If using the venting option, install the narrower 0.3 mm I.D. jet tube (PN 283168). Use the 0.8 mm I.D. jet tube (PN 319774) when operating without the venting option. To replace the jet tube, see Chapter 5, "Replacing the Jet Tube" on page 48.

4. Extend the column either 3.5 cm (nonventing) or 0.35 cm (venting option) above the top of the ferrule depending on whether or not the venting option is used (refer to Figure 3.22). Mark the column just beyond the 1/16" nut to verify proper column position.

5. Gently insert the column into the detector, maintaining the proper column position (Figure 3.22). Verify the column mark from step 4 is just below the nut. Tighten the 1/16" nut until the column can no longer be moved. Refer to Figure 3.23 for the correct column placement in the detector base.

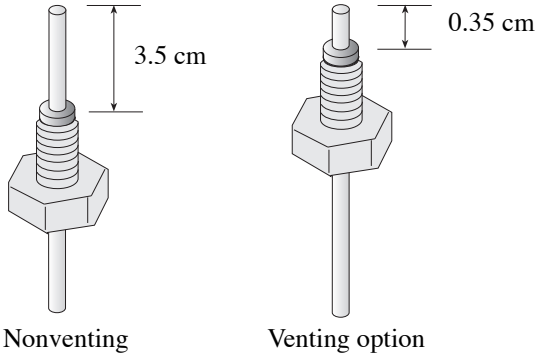


Figure 3.22. Positioning the ferrule on the column

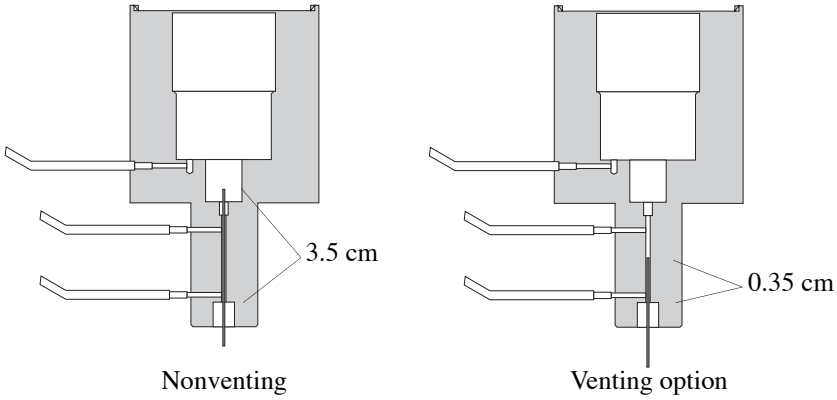


Figure 3.23. Column placement in the detector base

Installing the Venting Option

When installing the venting option (PN 319514), provide sufficient air supply pressure and flow for proper vent valve operation. When using an OIM gas flow module on the Agilent 7890 or 6890 GC, plumb the air supply through the OIM's air inlet and outlet, and set the flow rates between 300–500 mL/minute to provide the optimum flow rate of 20–30 mL/minute through the XSD during normal operation. Flow restrictors located inside the vent valve manifold make the high flow rate necessary. The Agilent 7890 or 6890's electronic pneumatics control (EPC) modules do not monitor actual flows (simply a calculated flow based on no backpressure or restrictors). Therefore, the flow rate must be set this high to deliver the working range of 20–30 mL/minute of air to the XSD. When the vent valves are activated, route the supply gas to a restrictor that then provides air into the detector base (150–200 mL/minute). At the same time, the vent valve switches and opens up the vent line to the vent port. This higher air flow through the jet line permits virtually 100% venting of the column effluent. Venting of an entire halogenated solvent peak is possible if the flows are set properly.

If no gas flow module is installed, well-regulated gas supplies must be provided because fluctuations in delivery pressure cause unstable detector responses.

Use the following procedure to install the XSD venting option:

1. Replace the 0.8 mm I.D. jet tube (PN 319774) with the 0.3 mm I.D. jet tube (PN 283168) included in the vent valve option startup kit. Refer to Chapter 5, “Replacing the Jet Tube” on page 48.

NOTE: Failure to use the correct jet tube (0.3 mm I.D.) with the venting option results in incomplete venting of the column effluent.

2. Mount the vent valve manifold to the top of the GC using the mounting screw (PN 252361). Make sure that the vent valve manifold is not too close to the reactor assembly. Select a location that minimizes heat transfer from the top of the GC to the vent valve manifold.

3. Route the gas supply lines from the XSD to the vent valve manifold (instead of the cross fitting used without the venting option) (refer to Figure 3.24 and Figure 2.1).

4. Using the 1/16" nut (PN 169640) and ferrule (PN 112433), connect the gas supply line leading from the pressure control manifold in the Agilent 6890 GC to the fitting on the vent valve manifold labeled “IN”.

5. Connect the proper gas lines from the detector base to the connections on the vent valve manifold labeled “VENT”, “WALL”, and “JET”. The gas lines are labeled appropriately. If the labels are not on the gas lines, refer to Figure 3.24 for proper identification.

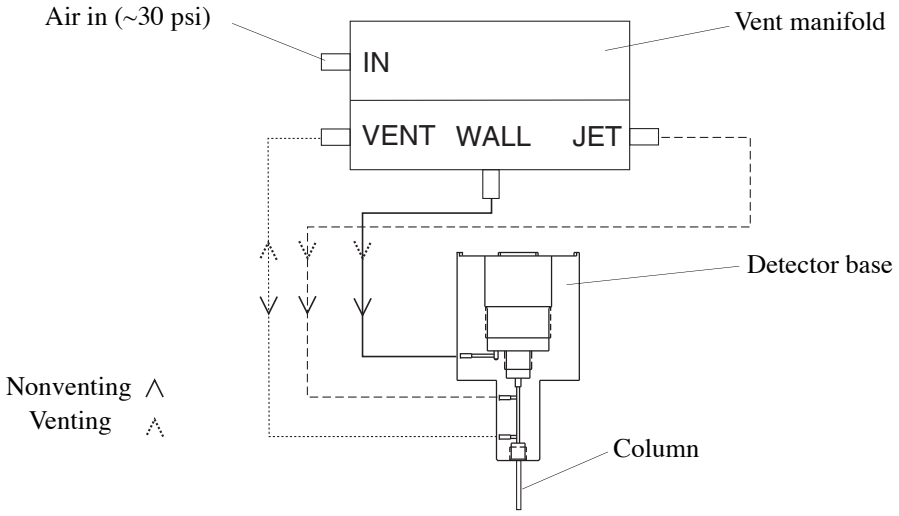


Figure 3.24. Gas connections between the XSD Detector Assembly and the vent valve manifold

6. Connect the two ends of the V-shaped vent cable (PN 264234) to the connectors on both the vent exit valve and the boost valve.

7. Connect the opposite end of the V-shaped vent valve cable to the valve five or valve six connector of the Agilent 6890 or 7890 GC's external events cable (PN 252569). Configure the selected valve (i.e., valve five or six) to a "switching" mode using the Agilent 6890 or 7890 GC's keypad. Refer to the *Agilent 6890 or 7890 Series GC Operator's Manual* for more details.

8. To vent column effluent, use the Agilent 6890 or 7890 GC's keypad to program the appropriate valve to the On position for the time period required in the analysis to vent the column effluent out of the detector base. Program the Off time in the analysis to return to normal operating conditions. A baseline disturbance or offset is normal during the time that the venting option is activated, caused by the significantly higher gas flows through the detector during this time.

9. Install the GC column (refer to "Installing the GC Column" on page 36 in this chapter) or install a no-hole ferrule in the bottom of the detector base where the column would be inserted.

10. Program the EPC module to read between 300–500 mL/minute of air. Attach the flow measurement adapter (PN 319631) to the adaptor fitting on the detector base. Using a gas flow meter, verify this setting provides a total air flow of approximately 20–30 mL/minute when the vent is closed. Adjust the EPC setting accordingly until 20–30 mL of air is measured from the flow measurement adapter.

11. Remove the flow measure adapter and verify the flow out of the jet tube is about half of the total air flow. This split does not have to be exactly equal, but there should be an adequate flow through each channel.

NOTE: When the vent valve is actuated, flow through the jet line increases significantly (>100 mL/minute), blowing the column effluent out of the vent valve.

12. If the reactor assembly is not installed, see steps 9–12 of "Installing the Model 5360A XSD on an Agilent 6890 or 7890 GC" on page 30 of this chapter.

13. Ensure the GC column is correctly positioned in the detector (refer to "Installing the GC Column" on page 36 of this chapter).

NOTE: When using the vent valve option, install the 0.3 mm I.D. jet tube (PN 283168). To replace the jet tube, see Chapter 5, "Replacing the Jet Tube" on page 48.

Installing the Model 5360 XSD onto the PID

The Model 5390 PID/XSD consists of the Model 5360 XSD mounted on the Model 4430 PID. Depending on GC, Table 3.1 or Table 3.2 summarizes the gas supply connections at the OIM pressure control manifold for the Model 5390 PID/FID. See the *Model 5360 Halogen Specific Detector Operator's Manual* for installating, operating, and troubleshooting the Model 5360 XSD. Also see the Model 5360 manual for installing electrical connections between the XSD and the Model 5300 Detector Controller.

Agilent 7890 or 6890 GC

1. Remove the attached fitting from the PID outlet on the side of the PID sensor.

2. Slide a ⅛" x 1⁄16" I.D. ferrule (PN 216366) onto the XSD jet tube (PN 245621) (Figure 3.25)

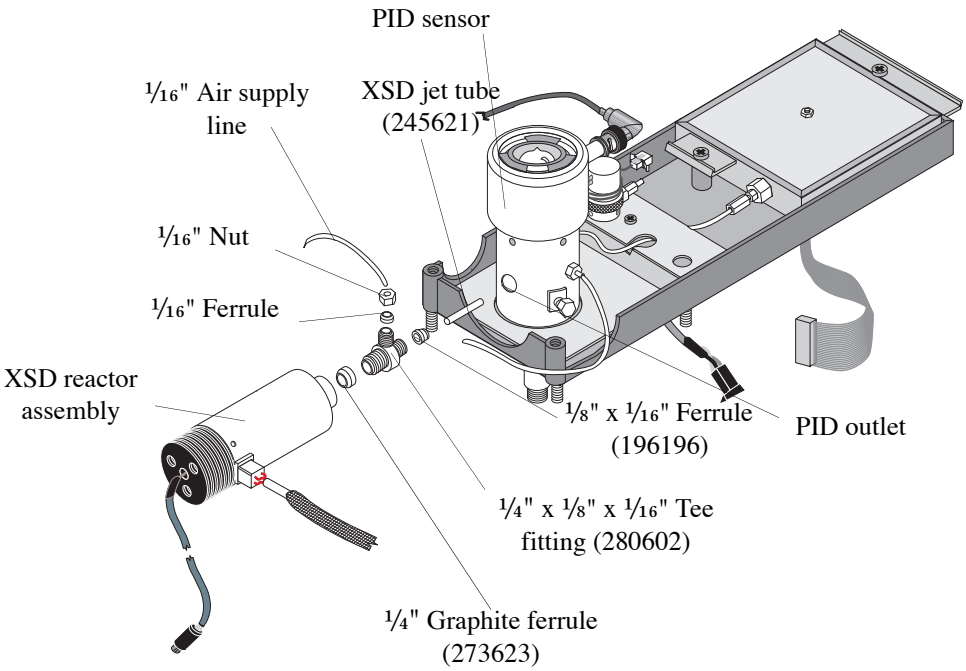


Figure 3.25. Model 5390 PID/XSD assembly

3. Place the jet tube and ferrule into the PID outlet, ensuring the jet tube enters the small recess in the back of the PID outlet.

4. Tighten the ⅛" end of the tee fitting (PN 280602) into the PID outlet. Apply light pressure to the jet tube to hold it in place while tightening.

5. Carefully slide the ¼" graphite ferrule (PN 273623) onto the ceramic tube in the base of the XSD reactor assembly. Ensure the tapered end of the ferrule faces toward the reactor.

6. Screw the XSD reactor assembly onto the ¼" side of the tee fitting. Tighten securely, but do not overtighten.

7. Remove the protective cap from the gas inlet marked “Air” on the front of the OIM pressure control manifold. Attach the air supply line to this inlet using a ⅛" brass nut and ferrule (not supplied). Refer to Figure 3.26 (7890) or Figure 3.17 (6890).

8. Ensure the gas line is pushed into the connector as far as it can go before tightening the nut onto the connector. Securely tighten each ⅛" nut using a 7⁄16" wrench.

Agilent 7890

1. Install the hoodlum assembly, provided with the OIM, in the Air position (refer to Figure 3.26).

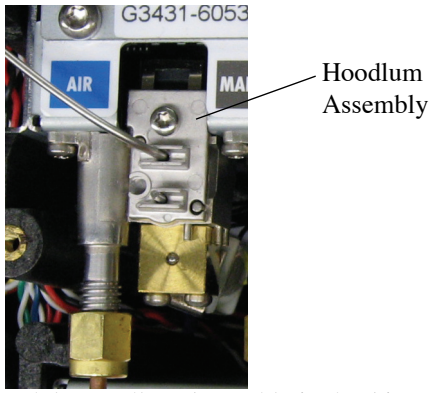


Figure 3.26. EPC Module Hoodlum Assembly in the Air Position

2. Cut the ⅛" nib off the end of the hoodlum assembly.

Agilent 6890

1. Cut a length of 1/16" stainless steel tubing (PN 193409, supplied in the detector gas kit) that is long enough to reach from the pneumatics compartment to the tee fitting between the PID and XSD. Route the tubing parallel to the PID makeup and sweep gas lines from the pneumatics compartment along the tubing guides on the top of the GC to the detector area.

2. Attach one end of the stainless steel tubing to the outlet marked “A” on the gas outlet block of the OIM pressure control manifold (refer to Figure 3.17) using a 1/16" nut (PN 223057) and ferrule (PN 216366).

3. Ensure the stainless steel tubing is pushed into the gas outlet block as far as it can go before tightening the nut into the gas outlet block. Securely tighten the nuts using a 5/16" wrench.

Agilent 7890 or 6890 GC

1. Slide the 1/16" nut from the tee fitting and a ferrule (PN 216366) onto the free end of the 1/16" stainless steel air line that extends from the OIM pressure control manifold. Ensure the tapered end of the ferrule faces into the nut.

CAUTION: Do not use H_2 as the carrier gas.

2. Turn on the gas supplies and verify all connections are leak tight. The gas supply requirements for the Model 5390 Tandem PID/XSD are presented in Table 3.4.

Table 3.4. Gas supply requirements for the Model 5390 PID/XSD

Gas Requirement	Species	Flow Rate
PID makeup gas	N_2 or He	10–20 mL/minute
PID sweep gas	N_2 or He	20–35 mL/minute
XSD reaction gas	Air	20–40 mL/minute

Refer to Chapter 4, “Operation” on page 33 in this manual and to the *Model 4430 Photoionization Detector Operator’s Manual* for detailed operating instructions.

Chapter 4 Operation

This chapter discusses Model 5360A XSD operation for analyzing samples.

Recommended Settings for General Operation

The following are typical settings for operating the XSD with the Agilent 6890 GC.

Base Temperature

- The heater cable, controlled by the GC, heats the detector base.
- Set the temperature to at least the maximum oven temperature used for the run. The optimal temperature is 20°C above the maximum oven temperature.
- The recommended maximum temperature for the heated base is 300°C. The maximum temperature should not exceed the recommended maximum column temperature.

Controller Gain

- Set the internal jumper on G3 in the Detector Controller (refer to Figure 4.1). G3 is the x10 setting; G2 is the x1 setting; and G1 is the x100 setting.

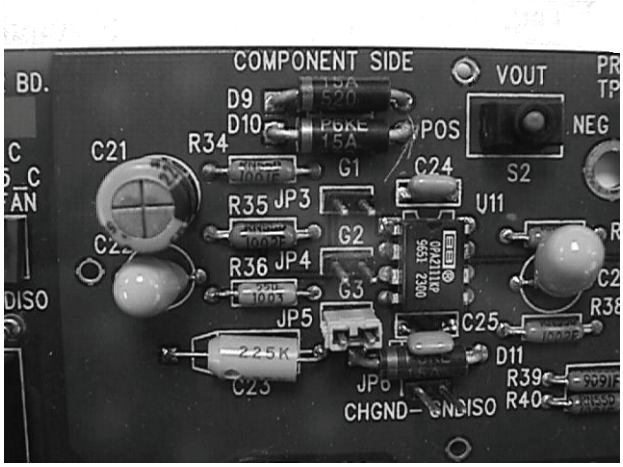


Figure 4.1. Close-up of the Model 5300 Detector Controller board

Output Signal

- Typically positive, 0–1 V DC

Oxidant Gas Flow

- 20–30 mL/minute air
- Typically split 15 mL/minute to jet, 15 mL/minute to reactor sweep (approximate)
- If the XSD is installed with the Model 4430 PID, use the gas flow shown in Table 3.4.

Reactor Temperature

- 1,000°C (set from the Detector Controller front panel)

General Operation

1. Verify the column, wall, and jet gas flows are set properly. Take measurements easily using the flow measurement adapter supplied with the XSD startup kit. See Chapter 3, “Installing the Model 5360A XSD” on page 30.

2. If the venting option is installed, verify vent flow occurs through the flow measurement adapter and the vent exit valve when both vent valves are activated.

3. After setting the proper flows and reinstalling the reactor assembly, turn on the Detector Controller power switch.

4. Wait for the reactor assembly to heat to the set point (10–15 minutes).

5. Once the reactor assembly has reached the selected temperature, verify the At temperature LED is on and remains stable.

6. Allow the detector to equilibrate for approximately 15–30 minutes.

7. Make an injection and observe that the baseline level peaks are upward. This confirms that the polarity is set correctly, the gain is at an acceptable level, and the power is being supplied to the reactor assembly.

8. When changing reactor temperatures, allow 15 minutes to reach the selected temperature and another 15 minutes to fully reach equilibrium.

9. Proceed with the chromatographic analysis.

Chapter 5 Maintenance

Chapter 5 describes maintenance of the Model 5360A XSD.

Replacing the Probe Assembly

1. Turn off the power to the Detector Controller. Allow at least one hour for the reactor assembly to cool to the point where it can be safely touched.

2. Disconnect the signal probe cable from the signal probe Molex connector on the top of the probe assembly by pushing the latch and the cable forward and gently pulling upward.

   WARNING: The probe assembly is hot when mounted on an actively heated reactor. Handling the probe assembly without allowing sufficient time for the reactor to cool to room temperature can cause serious burns.

3. Unscrew the three attachment screws, located on the top of the probe assembly, from the reactor cap.

4. Carefully remove the probe assembly from the top of the reactor. If it is not easily removed, check the attachment screws and ensure they are not attached to the reactor cap.

5. Remove the replacement probe assembly from its protective package. Install the attachment screws into the probe assembly.

6. Carefully install the new probe assembly into the reactor core.

7. Align the attachment screws and tighten into the reactor cap.

8. Connect the signal probe cable to the signal probe Molex connector.

9. Turn on the power to the Detector Controller.

10. Wait 30 minutes for the probe assembly to thermally stabilize. Monitor signal output until the output signal stops drifting down and stabilizes. After the signal is stable, begin chromatographic analysis.

Removing the Reactor Assembly From the Detector Base

1. Turn off the power to the Detector Controller. Allow at least one hour for the reactor assembly to cool to the point where it can be safely touched.

2. Lower the GC oven temperature.

3. Disconnect the signal probe cable from the signal probe Molex connector located on the top of the probe assembly.

WARNING: The reactor assembly may be hot. Handling the reactor assembly without allowing sufficient time for it to cool to room temperature can cause serious burns.

4. If replacing the reactor core, remove the probe assembly (refer to "Replacing the Probe Assembly" on page 45 in this chapter).

5. Disconnect the four-pin connector of the reactor power cable from the reactor core (refer to Figure 5.1).

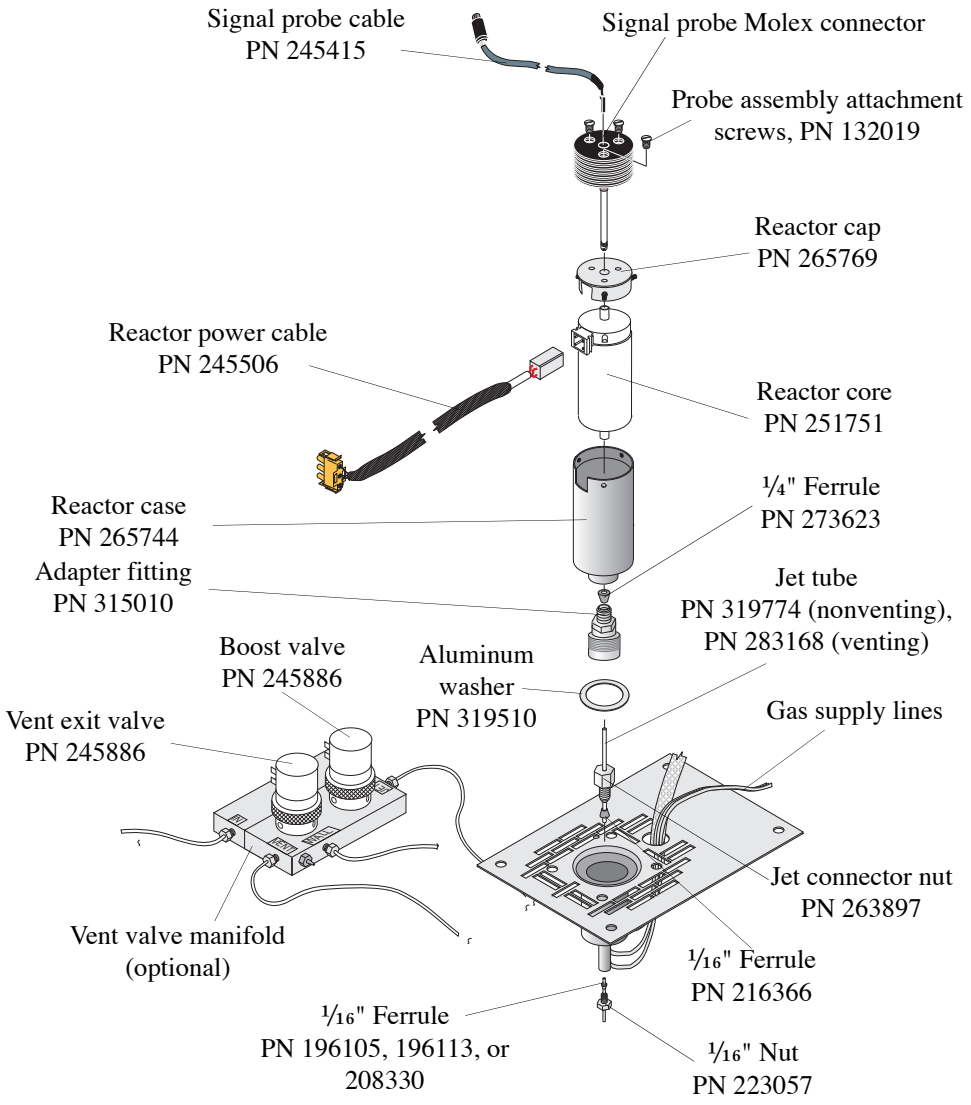


Figure 5.1. Model 5360A Detector Assembly with venting option

6. Carefully unscrew the reactor assembly from the detector base.

Replacing the Reactor Core

1. Allow the detector to cool to room temperature.

2. Remove the probe assembly (refer to “Replacing the Probe Assembly” on page 45 in this chapter).

WARNING: Any dust generated by sanding, filing, or machining the reactor core material is considered hazardous to your health. For more information, please reference MSDS No. 135 by the Carborundum Company, Fibers Division.

3. Remove the reactor assembly (refer to “Removing the Reactor Assembly From the Detector Base” on page 45 in this chapter).

4. Screw each set screw **into** the reactor cap using a 1/16" Allen wrench. Remove the reactor cap, making sure that the set screws do not fall out.

5. Invert the reactor case and push on the bottom of the reactor core, or use the four-pin socket to gently pull the reactor core out of the reactor base.

6. Remove the 1/4" graphite ferrule (PN 273623) from the bottom of the reactor case. Make sure that all the pieces of the ferrule are removed.

1. Install the new reactor core in the reactor case.

WARNING: Wash hands with warm soapy water immediately after handling the reactor core. If dust contacts eyes, immediately rinse with water and seek medical attention.

2. Reinstall the reactor cap using a 1/16" Allen wrench. Screw each set screw **out** of the reactor cap (counterclockwise) until it seats in the reactor case. **Do not overtighten**—the set screws should not be able to be pulled through the holes in the reactor case.

3. Install a new 1/4" graphite ferrule (PN 273623) onto the reactor core.

4. Reinstall the probe assembly (refer to “Replacing the Probe Assembly” on page 45 in this chapter).

5. Carefully screw the reactor assembly into the detector base, taking care not to bend the jet tube or crack the reactor core.

6. Reconnect the reactor power cable and the signal probe cable.

7. Turn on the power to the Detector Controller. Allow approximately 30 minutes for equilibration.

Replacing the Jet Tube

1. Remove the reactor assembly (refer to “Removing the Reactor Assembly From the Detector Base” on page 45 in this chapter).

2. Disconnect the GC column from the detector base.

WARNING: The jet tube may be hot. Handling the detector base without allowing sufficient time for it to cool to room temperature can cause serious burns.

3. Using the ½" nut driver tool (PN 319509C) supplied with the detector startup kit, unscrew the adapter fitting (PN 315010) from the detector base.

4. Using the 5⁄16" socket wrench (PN 246132) supplied with the detector startup kit, unscrew the jet connector nut (PN 263897) from the detector base. Gently remove the jet tube and fitting using needle-nosed pliers.

5. Replace the aluminum washer (PN 319510) if necessary, making sure not to scratch the bottom surface of the detector base.

6. If necessary, wipe the bottom surface of the detector base with a clean, methanol-moistened cotton applicator to remove any debris.

7. Using needle-nosed pliers, place the new 1⁄16" jet tube (PN 319774 or PN 283168 (venting option)) into the reactor base. Slide a 1⁄16" graphite-Vespel® ferrule (PN 216366) over the jet tube with the flat side down (refer to Figure 5.1). Slide the jet connector nut (PN 263897) over the jet tube. Gently tighten the jet connector nut using the 5⁄16" socket wrench. Do not overtighten.

8. Reattach the adapter fitting to the detector base using the ½" nut driver tool.

9. Reinstall the reactor assembly, taking care not to bend the jet tube.

10. Reinstall the GC column. See Chapter 3, “Installing the GC Column” on page 36.

11. Reconnect the reactor power cable and the signal probe cable.

12. Turn on the power to the Detector Controller. Allow 30 minutes for equilibration.

Chapter 6 Troubleshooting

Once the Model 5360A XSD is installed and operating properly, note for future reference the signal display, baseline offset, noise levels, and relative sensitivity at the various reactor temperatures.

The following chart lists the most common problems that can occur when using the XSD, along with their corresponding corrective actions. Each problem potentially may be caused by more than one reason. Before using this guide, please become thoroughly familiar with the operating and maintenance information contained in previous chapters. If a problem still exists after reviewing the following chart or if a particular problem is not addressed, contact OI Analytical Technical Support at (800) 336-1911 (USA/Canada) or (979) 690-1711 for assistance.

Symptom	Corrective Action
High baseline	Disconnect the signal probe cable. If the baseline does not go to zero, check the chart recorder or data acquisition connectors. Reconnect the signal probe cable. Verify the reactor temperature has not been set to a higher temperature. Cap the column inlet with a no-hole ferrule. If the baseline returns to normal, check the column and injector for contamination. Check the gain and voltage range jumpers on the Detector Controller board, located inside the Detector Controller, to ensure they have not been changed (typical gain = x10 (G3) and JP7 (1VOUT) installed). Allow time for the system to stabilize.

Symptom	Corrective Action
Low baseline	If the baseline is less than zero, verify the polarity jumper has not been changed (the standard setting is inverted). Check the connections to the signal probe cable at both the probe and DIN connectors. Check the gain jumper to ensure it has not been changed (typical gain = x10 (G3) and JP7 (1VOUT) installed). Verify the reactor temperature has not been set to a lower temperature. Check that the oxidant and column flow rates are properly set. Check for degraded seals. Check for leaks from the gas supply lines to the detector and vent valves.

Symptom	Corrective Action
No or low response	Verify the power is on. Check for column continuity, column head pressure, and septum seal quality. Check the oxidant flow rates. Check the signal probe and reactor cable connections. Increase the reactor temperature. If no change in baseline is observed, check the data display and acquisition cables and their connections. Check the vent timing values. Check the vent valve for improper seating or sealing. Check the oxidant flow rates and verify the sweep oxidant gas flow is not obstructed. Check if the jet tube is bent. Check the injection technique. Check for unswept dead volume in the injector. Check for cold spots between the column and the detector assembly. Check for foreign matter in the detector base and reactor assembly. Check for flow through the jet tube to ensure there is no blockage in the column or at the jet ferrule. Check the probe assembly.

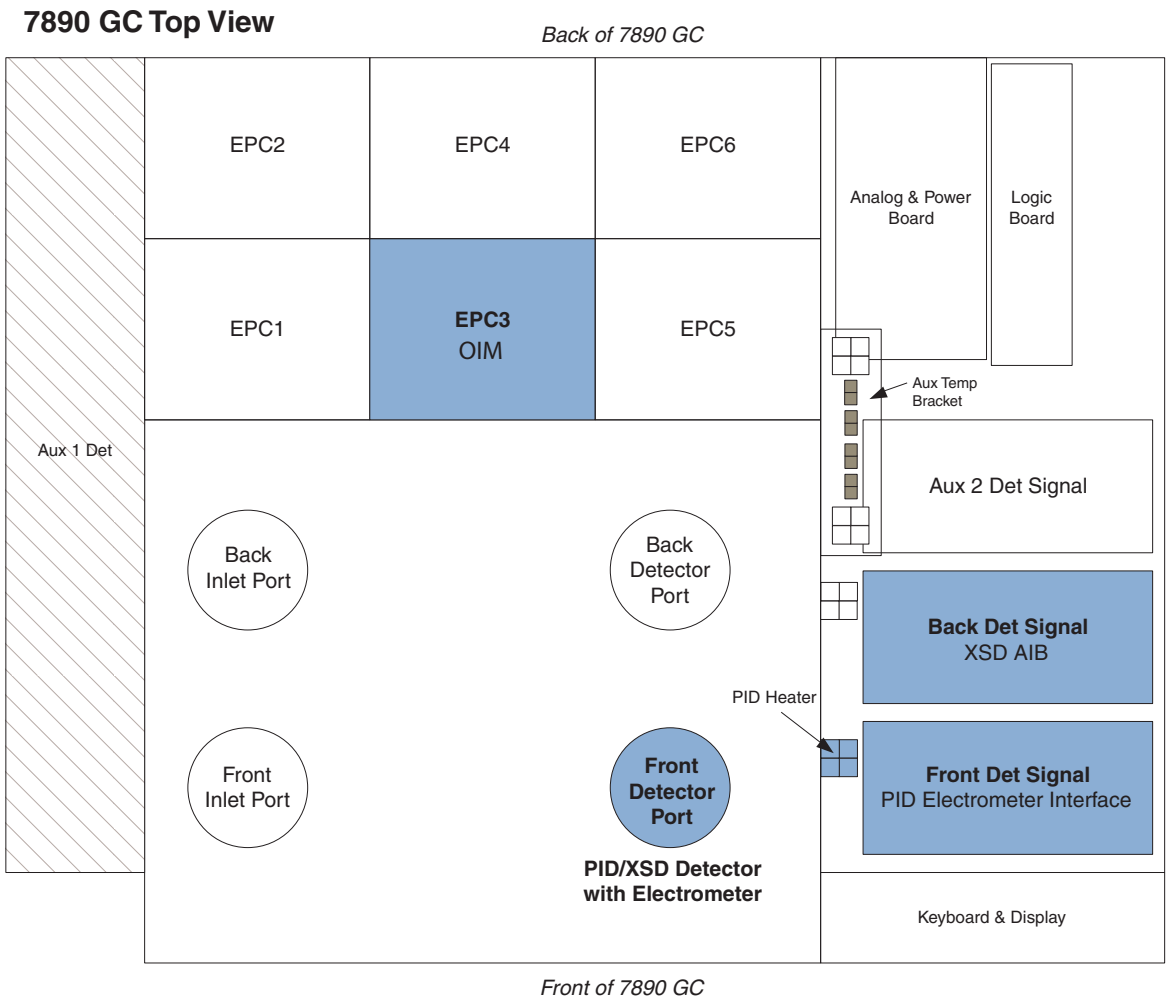
Chapter 7 Replacement Parts

This chapter provides a list of replacement parts and support items for the Model 5360A XSD and its associated options. An asterisk indicates replacement parts that are considered expendable (XPN). Replace expendable parts regularly, since they may become deformed or broken. Keep a supply of expendable parts in stock.

Product	Size	Unit	PN	XPN
Adapter fitting	—	each	315010	
Adapter removal tool	1/2" nut driver	each	319509C	
Allen screw set	6-32, 1/8"	each	131961	*
Cross fitting	1/16"	each	319519	
Dual vent valve cable	—	each	264234	
External events cable	—	each	252569	
Ferrule	1/8" to 1/16" tube	10/pk	320828	*
Ferrule, graphite, for PID/XSD	1/16" tube	10/pk	204693	*
Ferrule, graphite, for PID/XSD	1/4"	10/pk	320827	*
Ferrule, graphite, for XSD	1/4"	each	273623	*
Ferrule, graphite/Vespel	1/16" x 0.8 mm I.D.	10/pk	196105	*
Ferrule, graphite/Vespel	1/16" x 0.4 mm I.D.	10/pk	208330	*
Ferrule, graphite/Vespel	1/16" x 0.5 mm I.D.	10/pk	196113	*
Ferrule, graphite/Vespel	1/16" x 0.0 mm I.D.	10/pk	197079	*
Ferrule, graphite/Vespel, for XSD	1/16" tube	10/pk	216366	*
Ferrule, stainless steel	1/16"	each	112433	
Flow measurement adapter	—	each	319631	
Gas outlet block for OIM gas flow module 6890	—	each	251785	
Jet connector nut	5/16"	each	263897	*
Jet removal tool	5/16" nut driver	each	246132	
Jet tube, nonventing	0.8 mm I.D. x 1.56"	each	319774	*
Jet tube, venting	0.3 mm I.D. x 1.56"	each	283168	*

Product	Size	Unit	PN	XPN
Nut, male, stainless steel	1/16"	each	223057	
OIM gas flow module 6890	—	each	285049	
Power cord for North America, 110 V	—	each	116038	
Probe assembly	—	each	251744	
Probe to controller cable	—	each	245415	*
Reactor assembly	—	each	273615	
Reactor base bracket for Agilent 6890 GC or 7890	—	each	280735	
Reactor cap	—	each	265769	
Reactor case	—	each	265744	
Reactor core	—	each	251751	
Reactor power cable from Model 5300 Detector Controller	—	each	245506	
Screw, stainless steel, machine	4-40 x 3/8"	each	132019	*
Startup kit for Model 5360A XSD	—	each	319518	
Tee fitting for Model 5390 PID/ XSD	1/16 x 1/8 x 1/4"	each	280602	*
Tubing, stainless steel	1/16" x .030 I.D.	foot	193409	
Vent boost valve, two-way (24 V)	—	each	245886	
Vent exit valve, two-way (24 V)	—	each	245886	
Vent valve manifold with valves	—	each	319515	
Venting option	—	each	319514	
Washer, aluminum		each	319510	*
Analog input board AIB, 6890			274886	
Analog Input Board AIB, 7890			324764	
OIM gas flow module, 7890			324768	
AUX EPC Module, 7890			324769	

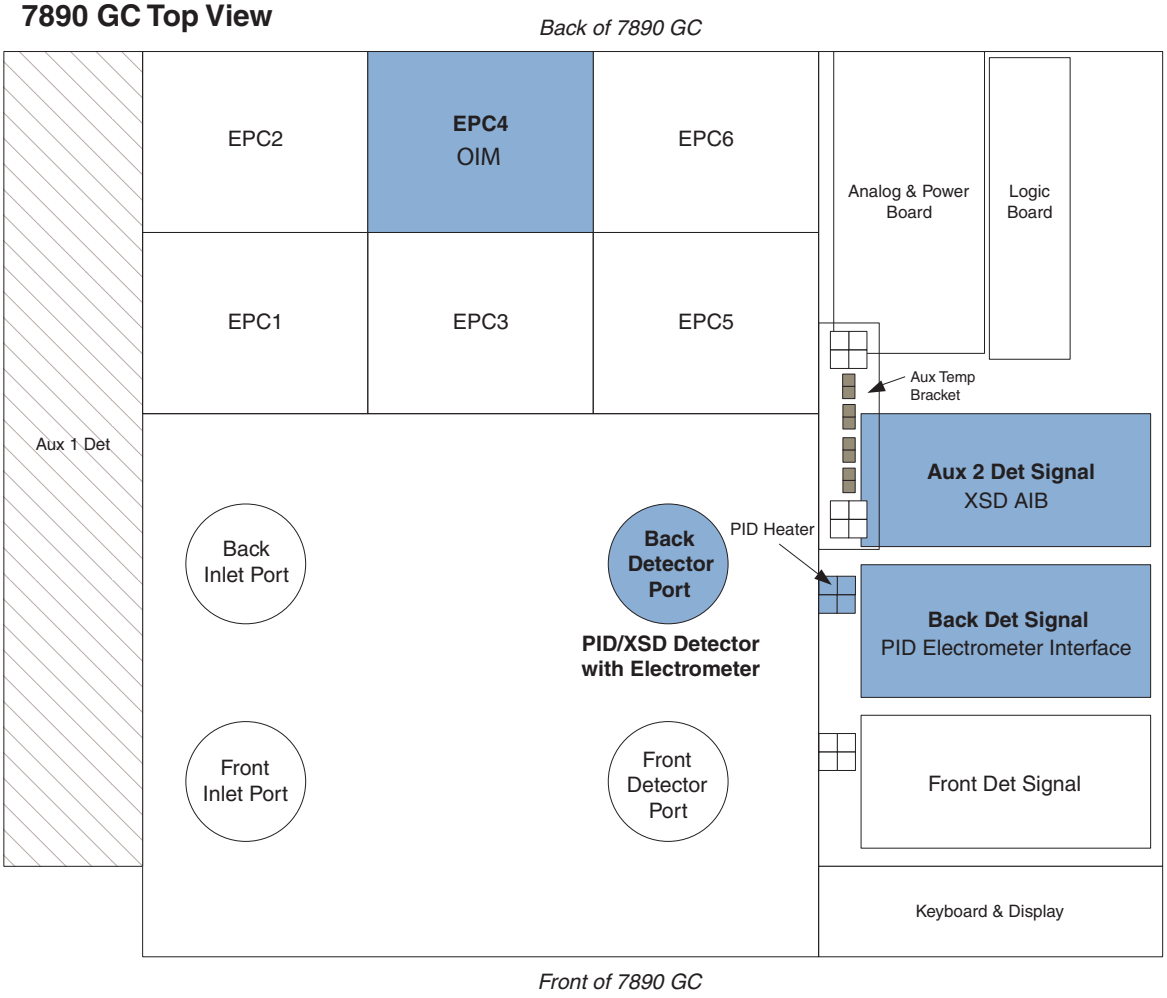
PID/XSD Configurations



Agilent 7890 GC Detector Configuration Selections
Front Det: CPDET FID, Htr, EPC
Aux Det 2: AIB

Figure 7.1. PID/XSD - Front Detector Configuration

7890 GC Top View



Agilent 7890 GC Detector Configuration Selections
Back Det: CPDET FID, Htr, EPC
Aux 2 Det: Install AIB with No Heater
OR
Back Det: CPDET FID&AIB, Htr, EPC

PID/XSD - Back Detector Configuration

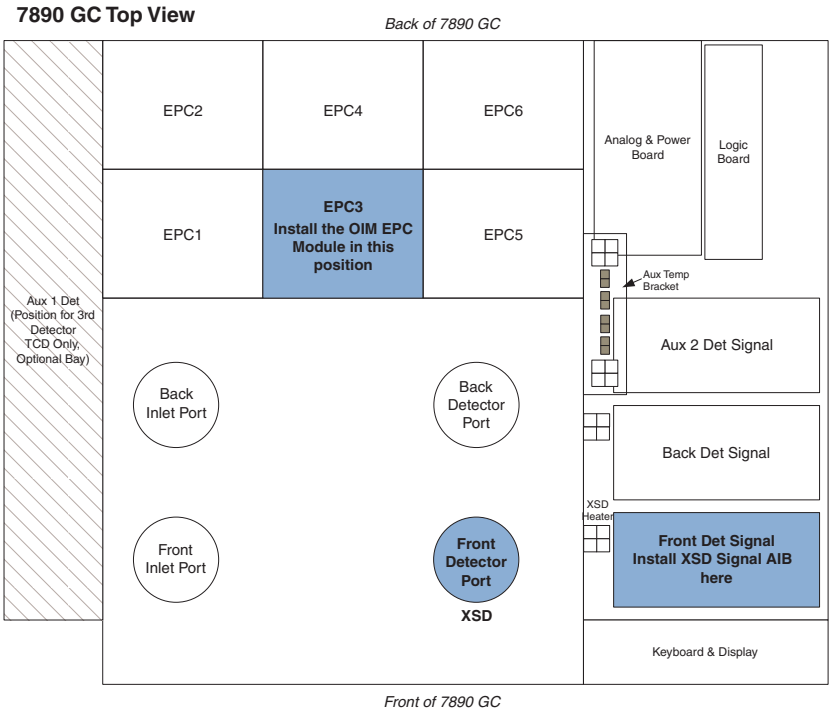
Chapter 8 Appendix

Location of Detectors, Modules, and Heaters for an Agilent 7890

Use the following figures and table to determine the appropriate location of the various components on an Agilent 7890.

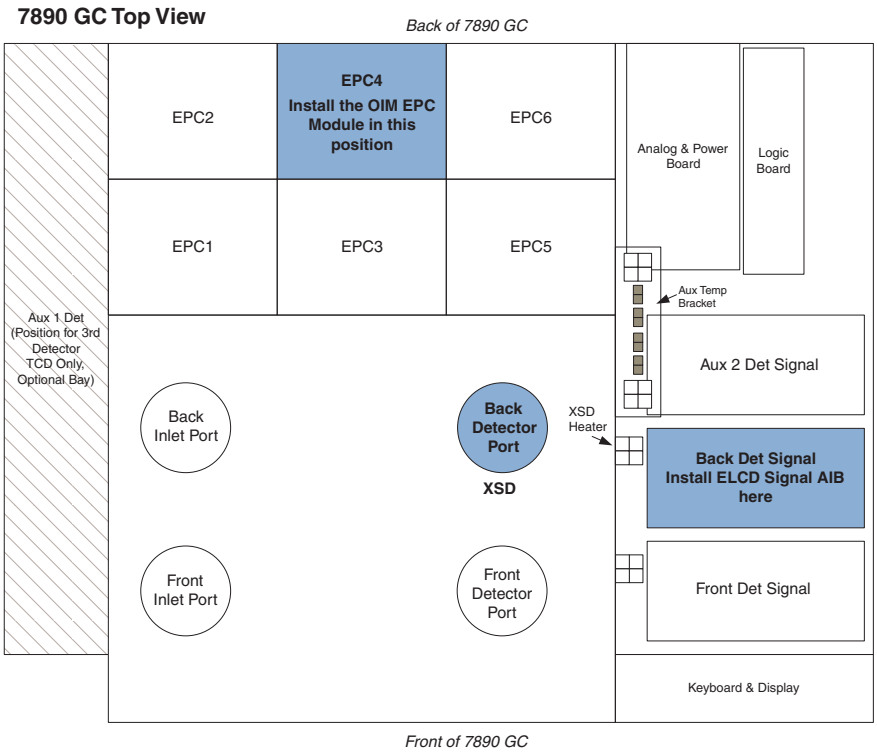
NOTE: These figures and table represent the *recommended* configurations. Other configurations are possible; refer to the *Agilent 7890 Operator's Manual* for other configurations.

XSD Standalone Recommended Configurations



Agilent 7890 GC Detector Configuration Selections
Front Det: CPDET AIB, with Htr, EPC

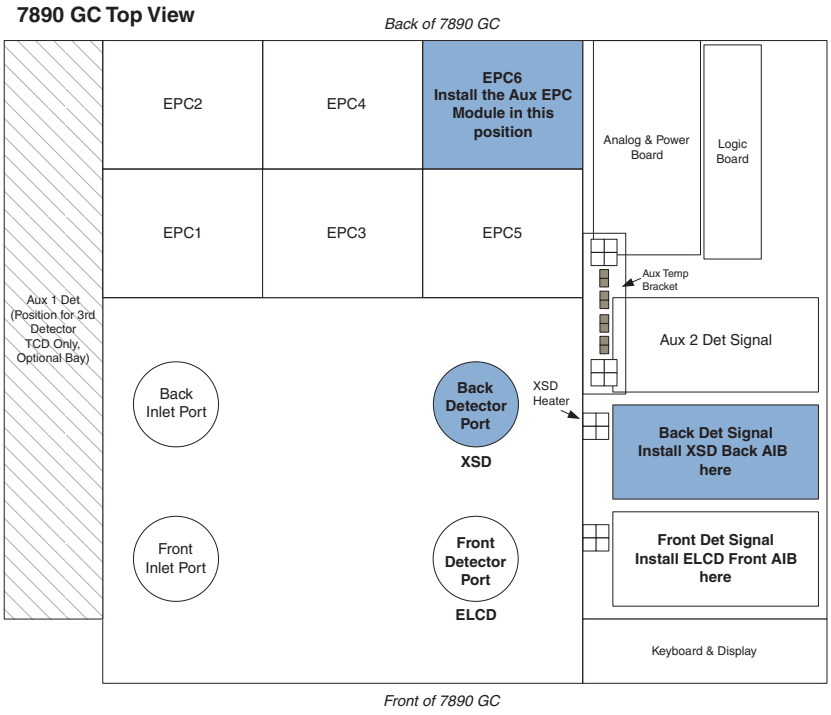
Figure 8.1. Agilent 7890 Top Detector Location for Standalone XSD - Front Position



Agilent 7890 GC Detector Configuration Selections
Back Det: CPDET AIB, with Htr, EPC

Figure 8.2. Agilent 7890 Top Detector Location for Standalone XSD - Back Position

Dual XSD Recommended Configuration with Aux EPC



Agilent 7890 GC Detector Configuration Selections
Back Det: CPDET AIB, No Htr, No EPC
Aux EPC: Pressure Aux 1,2,3
Chan 1 Gas Type Air
AUX Temp: Thermal AUX 1

Figure 8.3. Agilent 7890 Top Detector Location for XSD with an Aux EPC

PID/XSD Configurations

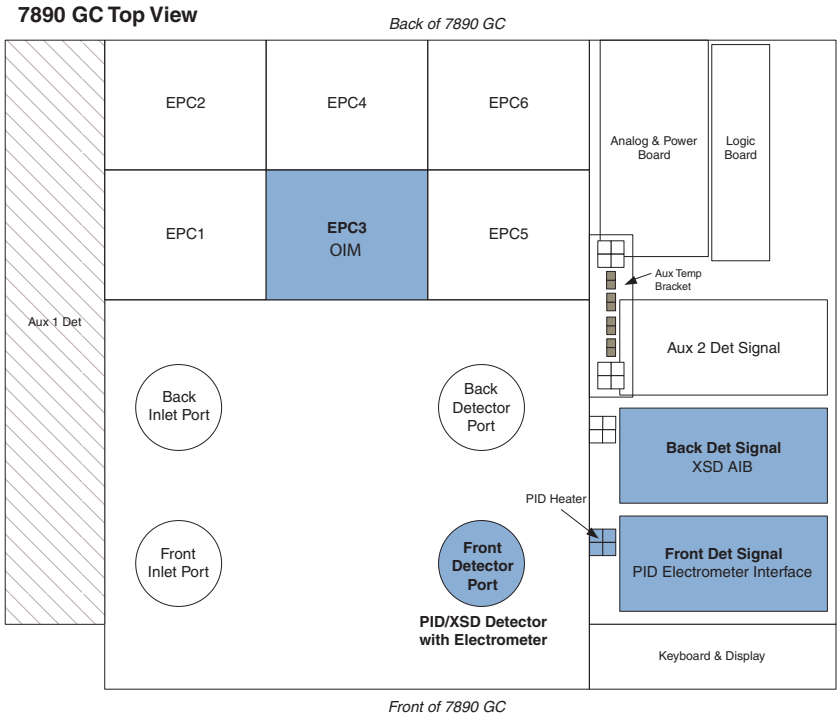
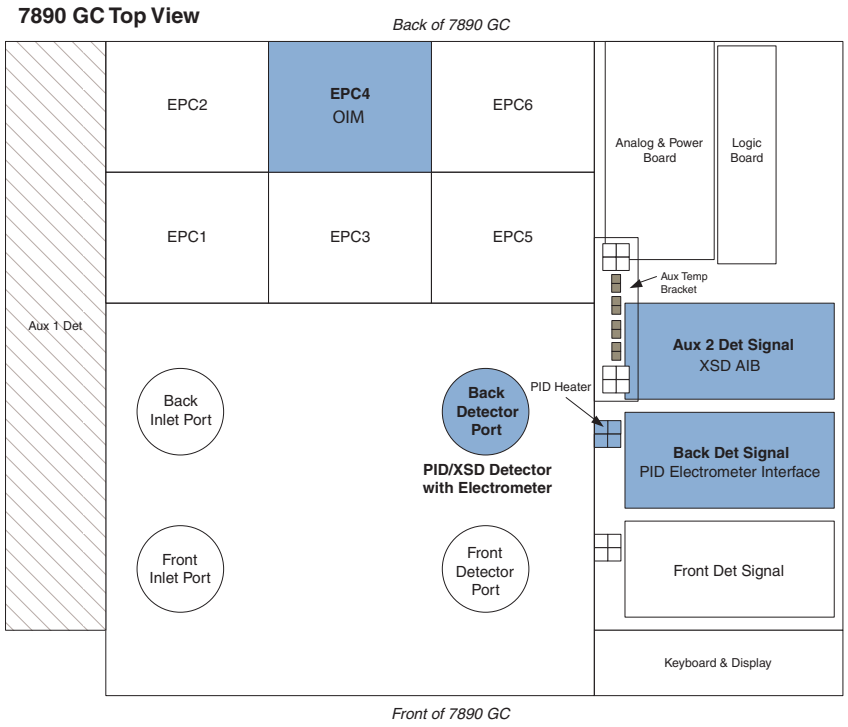


Figure 8.4. PID/XSD - Front Detector Configuration



Agilent 7890 GC Detector Configuration Selections
Back Det: CPDET FID, Htr, EPC
Aux 2 Det: Install AIB with No Heater
OR
Back Det: CPDET FID&AIB, Htr, EPC

Figure 8.5. PID/XSD - Back Detector Configuration

Generic Agilent GC Layout

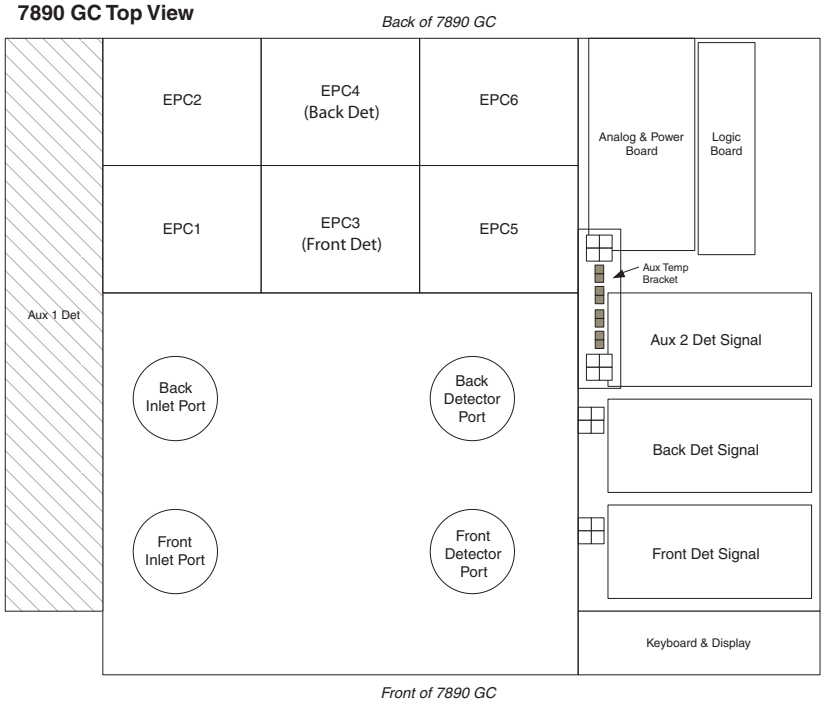


Figure 8.6. Agilent 7890 Top Detector Locations

Table 8.1. Agilent 7890 Configurations and Settings for OI Analytical Detectors

OI Analytical Detector Configuration	Hardware Installed in Front Det				Hardware Installed in Back Det				Hardware Installed in Aux 2 Det				Detector Configuration
	FID Electrometer Set	AIB	OIM Flow Module	Heater	FID Electrometer Set	AIB	OIM Flow Module	Heater	FID Electrometer Set	AIB	Aux EPC Flow Module	Heater	
ELCD Standalone (Figure 8.1)		X	X										Front Det: CPDET AIB, No Htr, EPC
ELCD Standalone (Figure 8.2)						X	X						Back Det: CPDET AIB, No Htr, EPC
ELCD w/Aux EPC (Figure 8.3)		X									X		Front Det: CPDET AIB, No Htr, No EPC Aux EPC: Pressure Aux 1, 2, 3 Chan 1 Gas Type Hydrogen
Dual ELCD with Aux EPC (Figure 8.3)		X				X					X		Front Det: CPDET AIB, No Htr, EPC Back Det: CPDET AIB, No Htr, EPC Aux EPC: Pressure Aux 1, 2, 3 Chan 1 Gas Type Hydrogen Chan 2 Gas Type Hydrogen
Dual ELCD with OIM (Figure 8.5)		X	X			X	X						Front Det: CPDET AIB, No Htr, EPC Back Det: CPDET AIB, No Htr, EPC
Tandem PID/ELCD (Figure 8.6)	X		X	X		X							Front Det: CPDET FID, Htr, EPC Aux Det 2: AIB
Tandem PID/ELCD (Figure 8.7)					X		X	X		X			Back Det: CPDET FID, Htr, EPC Aux Det 2: AIB **OR** Back Det: CPDET FID&AIB, Htr, EPC

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