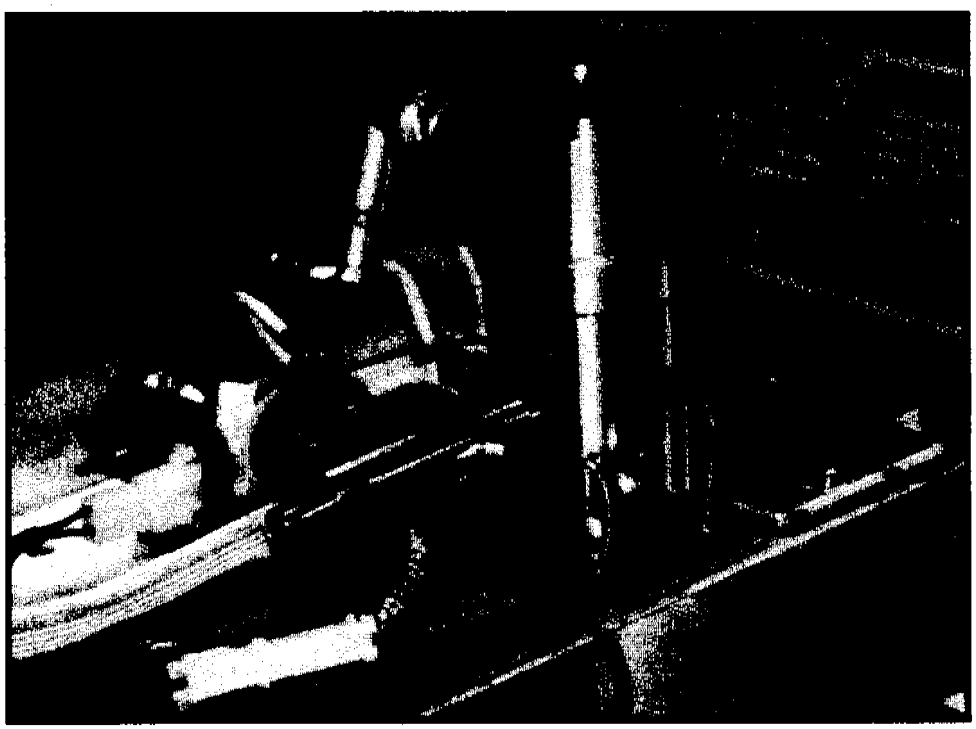
Model 4410 Flame Ionization Detector
Operator's Manual



O·I·Analytical

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Registration Card

Model 4410 Flame Ionization Detector

Operator's Name Company Name

Address City State Zip

Phone ( ) Date Received

Applications for which you use this equipment:

Serial #

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Chapter 1
Introduction

The OI Analytical Model 4410 Flame Ionization Detector (FID) is a hydrocarbon detector, specifically designed for the Hewlett-Packard (HP) Model 5890A and 5890 Series II GCs. The FID measures a sample's hydrocarbon content by using the flame ionization technique. Flame ionization is a destructive process in which hydrocarbon in a sample stream is combusted and ionized in a hydrogen-air flame, then the ionized products pass through an annular electrode. The ion current produced is measured by an electrometer circuit that outputs a voltage proportional to the amount of analyte in the sample.

The 4410 FID is designed to work in conjunction with the OI Analytical Model 4430 Photoionization Detector (PID). The FID mounts to the exhaust port of the PID, forming a dual-detector system for use in EPA municipal and industrial wastewater Methods 603, 604, and 609; EPA solid waste Methods 8015, 8030, 8040, 8060, 8090, and 8100; Benzene/Toluene/Xylene (BTX) analysis; underground storage tank monitoring; chemical waste testing; petrochemical analysis; flavors and fragrances; and pharmaceutical applications.

Features

- Attaches to the exhaust port of the PID, forming a dual-detector set that occupies only one detector port on a GC without the use of transfer lines or nonstandard fittings. This patented (U.S. Patent #4804846) detector design:
    1. Minimizes dead volume and eliminates cold spots in transfer lines.
    2. Allows use of the PID hydrogen sweep gas as the FID combustion gas.
    3. Requires the use of only one injector, one column, and one makeup gas.
    4. Greatly simplifies interfacing between detectors.
- Can be fully integrated into the GC design by using the available GC gas supplies, ignitor circuit, and electrometer, enhancing compatibility of components as a system and simplifying installation.
- Can be operated simultaneously with the PID, or either detector can be operated independently.
- Has both an electronic ignitor and a built-in port for flow checking.
- Incorporates an inert glass-lined stainless steel flame jet and a stainless steel collector electrode that are easily removed for inspecting and/or servicing.

Principal Applications

The FID responds to a wide variety of compounds. Common applications include:

EPA 603	Hydrocarbons (BTX)
EPA 604	Pesticides
EPA 609	Foods and flavors
EPA 8015	Consumer products
EPA 8030	Pharmaceuticals
EPA 8040	Underground storage tanks
EPA 8060	Chemical wastes
EPA 8090	Air toxins
EPA 8100	Total petroleum hydrocarbons
Petrochemical	Acids and bases
Carbohydrates	Alcohols
Solvents	Amines

Principle of Operation

The sample stream flows from the side port of the PID into the jet of the FID. The sample stream is combusted and ionized in a hydrogen-air flame. The ionized products pass through a negatively-biased annular electrode, producing an ion current. The ion current is converted to a proportional voltage output by an electrometer circuit. The voltage output is a direct representation of the amount of analyte in the carrier stream, i.e., the FID generates a response that is mass dependent.

Lower Detectability Limit:	5 pg carbon/sec (S/N = 3)
Signal/Noise:	3 at 5 pg/sec
Linear Dynamic Range:	$>10^6$
Sensitivity:	22 mCoul/g for propane
Maximum Linear Level:	100 ug benzene
Maximum Operating Base Temp:	270° C (limited by PID maximum)
Jet Tip:	0.20" I.D.
Materials of Construction:	Jet — Inert Glass-Lined Stainless Steel Collector — Stainless Steel Body — 316 Stainless Steel Ferrules — Vespel®
Gas Requirements:	H_2 — 99.999; 35 ± 2 ml/min He/N_2 — 99.999; 30 ± 2 ml/min (as carrier or makeup) Air — Dry; best available; 165 ± 15 ml/min

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.*

NOTE: Specific gas treatment strategies (including the proper selection of carrier and support gases) and gas traps significantly affect the sensitivity, accuracy, and precision of chromatography results. Trace impurities of oxygen and water can lead to column deterioration. Hydrocarbon impurities can affect FID sensitivity and can cause baseline irregularities. For optimal performance, use high capacity gas traps in conjunction with the specified high purity gases. OI Analytical offers the following line of high capacity gas traps:

Activated Charcoal Trap (OI Analytical #196782): S-shaped filter for trapping hydrocarbon impurities. Contains 30/60 mesh charcoal. Can be regenerated.

Molecular Sieve Trap (OI Analytical #196790): S-shaped filter for trapping water. Contains 45/60 mesh molecular sieve. Can be regenerated.

General Purpose Trap (OI Analytical #197848): Stainless steel scrubber for removing hydrocarbons, water, and oxygen. Contains a charcoal/molecular sieve/ambersorbe mixed bed. Cannot be regenerated.

Safety Notes

The instructions for installation and operation given in this manual are believed to be a thorough account for proper and safe operation. However, it is the responsibility of each laboratory to maintain the instrument in a condition suitable for safe use. Guidelines for maintenance are given later in this manual (see Chapter 5).

- 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.
- Turn the GC and detector power OFF and disconnect all line power when installing or servicing the equipment.
- 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.
- The FID contains a hydrogen-air flame. Keep all flammable and loose materials away from the detector. Do not lean over the FID to see if the flame is lit. Turn OFF all gas flows when servicing the detector.
- Wear safety glasses when using compressed gas. It is recommended to wear safety glasses at all times when working with the GC and/or the detector.

- Hydrogen is extremely flammable and has been identified as an asphyxiant. This gas and the cylinder containing this gas 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 their cylinders 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.

- Oxygen has been identified as an oxidizer. This gas and the cylinder containing this gas 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 the pure gas except under approved, controlled conditions by the analyst. The analyst should also avoid prolonged exposure to high concentrations of this gas.

Chapter 2
Description of Components

This chapter defines and describes the functions of the various components of the FID. Each significant component is identified in Fig. 2.1.

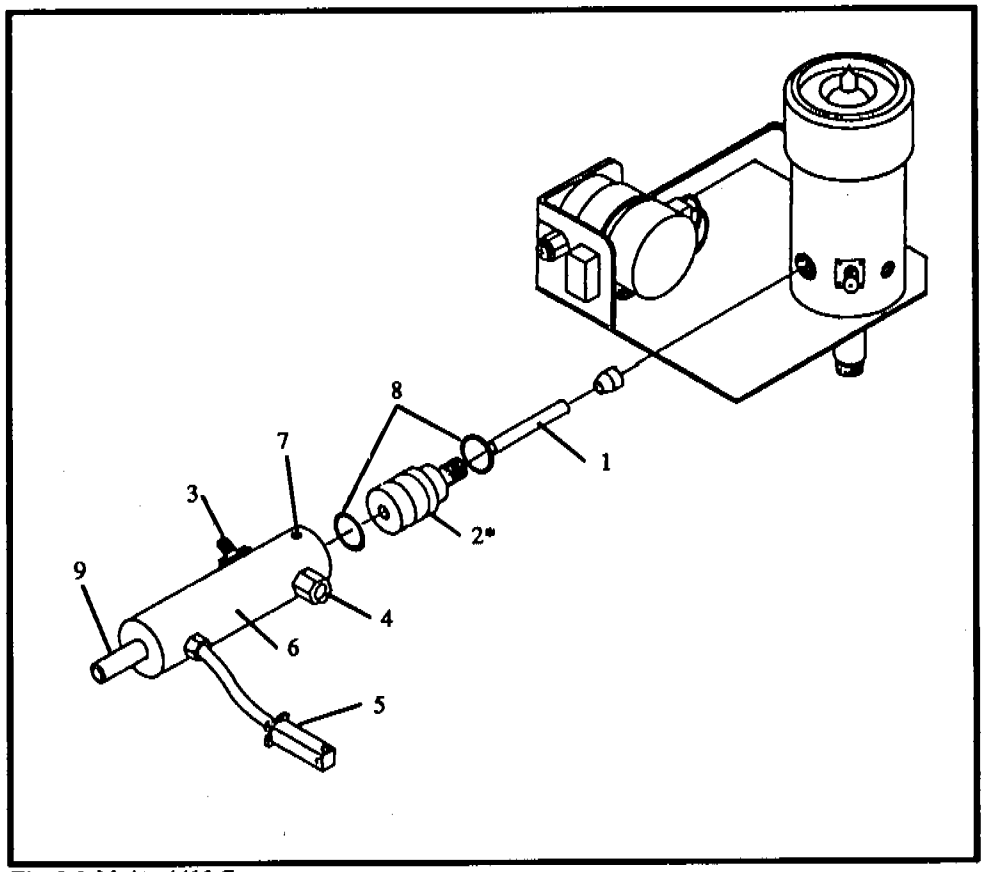


Fig. 2.1 Major 4410 Components

Detector Components

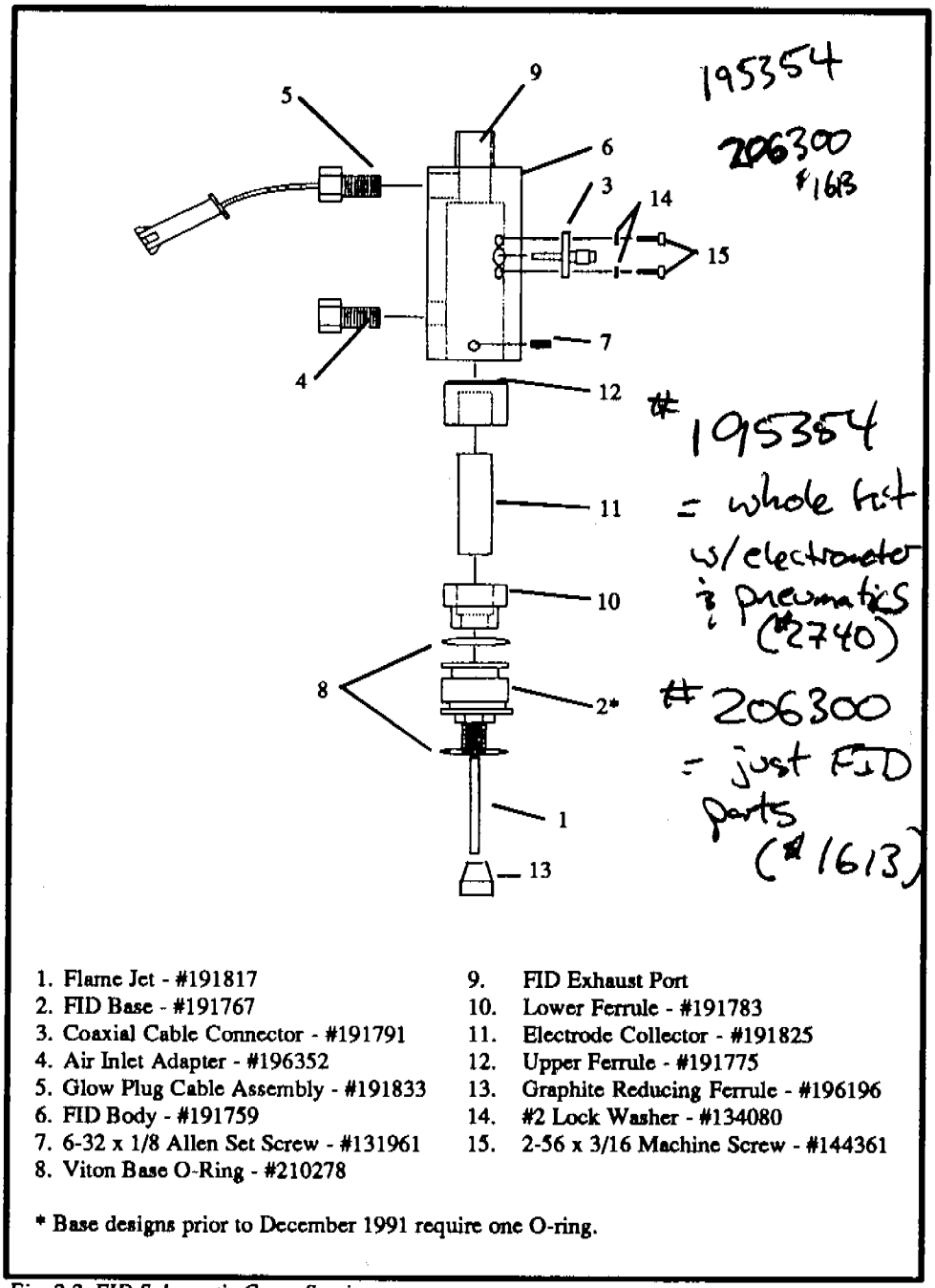
1. **Jet** (OI Analytical #191817) contains the sample as it flows from the PID into the FID. A hydrogen-air flame burns and ionizes the analyte at the end of the jet. The jet is composed of an inert glass-lined stainless steel tube.

2. **Base** (OI Analytical #191767) secures the FID to the PID. The base screws into the PID exhaust port and seals the jet to the PID with a 1/8" x 1/16" reducing graphite ferrule.

* Base designs prior to December 1991 require one O-ring.

3. **Coaxial Cable Connector** (OI Analytical #191791) connects to the metal outer wall of the annular collector electrode. The ion current generated in the FID is amplified by the electrometer circuit. A coaxial electrometer cable attaches here to transmit the ion current generated in the FID to the electrometer circuit.

4. **Air Inlet Fitting** (OI Analytical #196352) receives a 1/16" ferrule-type compression seal on the end of the FID air line. Air enters the FID and flows around the jet, supporting the combustion of hydrogen to produce a flame at the tip of the jet.



1. Flame Jet - #191817
2. FID Base - #191767
3. Coaxial Cable Connector - #191791
4. Air Inlet Adapter - #196352
5. Glow Plug Cable Assembly - #191833
6. FID Body - #191759
7. 6-32 x 1/8 Allen Set Screw - #131961
8. Viton Base O-Ring - #210278
9. FID Exhaust Port
10. Lower Ferrule - #191783
11. Electrode Collector - #191825
12. Upper Ferrule - #191775
13. Graphite Reducing Ferrule - #196196
14. #2 Lock Washer - #134080
15. 2-56 x 3/16 Machine Screw - #144361

* Base designs prior to December 1991 require one O-ring.

Fig. 2.2 FID Schematic Cross Section

5. **Glow Plug Cable Assembly** (OI Analytical #191833) ignites the FID flame. When the ignition button on the GC is pushed, a voltage provided by the GC power supply causes the glow plug to become hot enough to ignite the hydrogen-air mixture inside the FID. A soft "pop" may be heard when the flame lights. A loud sound indicates that excessive hydrogen may be present or that there may be insufficient air.

6. **Body** (OI Analytical #191759) contains and supports the internal components of the FID. It fits over the FID base and is secured by a set screw. The base is sealed by a set of O-rings. Fig. 2.2 shows the mechanical relationship of the various parts within the body.

7. **Set Screw** (OI Analytical #131961) keeps the FID body in place. It is not used to exert force on any seals and ***should not be over-tightened.***

8. **O-rings** (OI Analytical #210278) form a seal between the FID body and the base. The seal is secured by the set screw.

9. **Exhaust Port** vents the combustion products produced by the hydrogen-air flame in the FID.

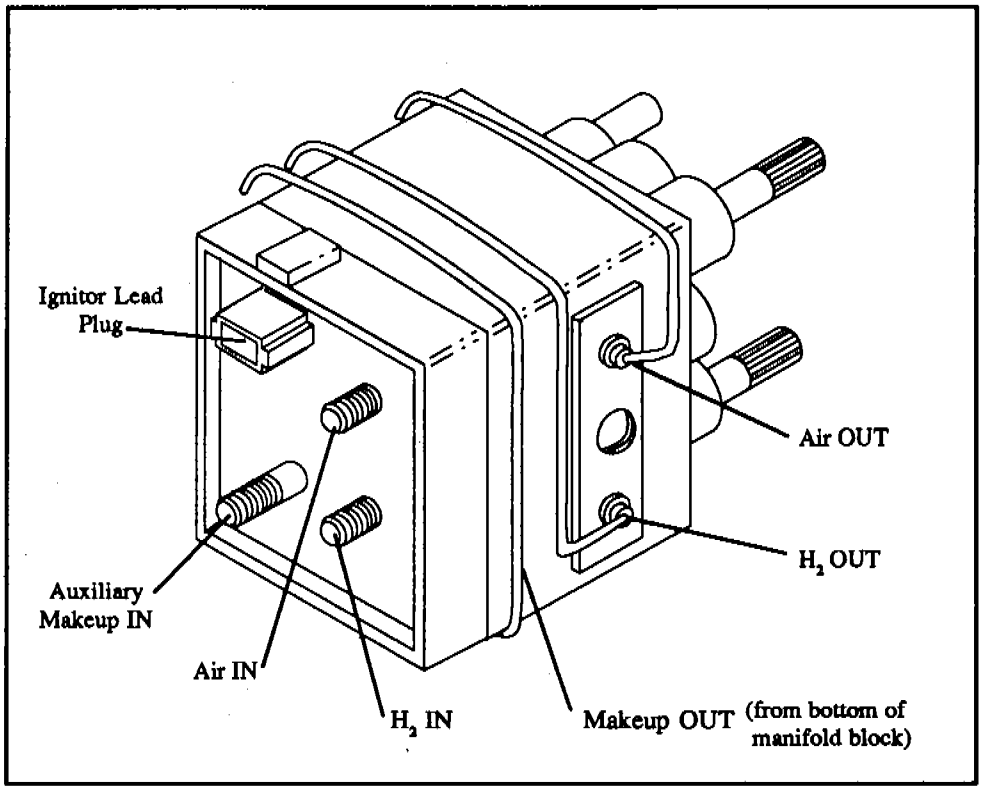


Fig. 2.3 PID/FID Gas Flow Module

Gas Flow Module (OI Analytical #192013). PID/FID installations and FID retrofit installations on HP 5890 Series GCs require the special Gas Flow Module (see Fig. 2.3) that is installed in the GC mainframe. The function of the Module is to receive, restrict, control, and distribute gas flows for hydrogen, auxiliary makeup gas, and air to the PID/FID detector set. The ignitor switch is also conveniently located on the Gas Flow Module.

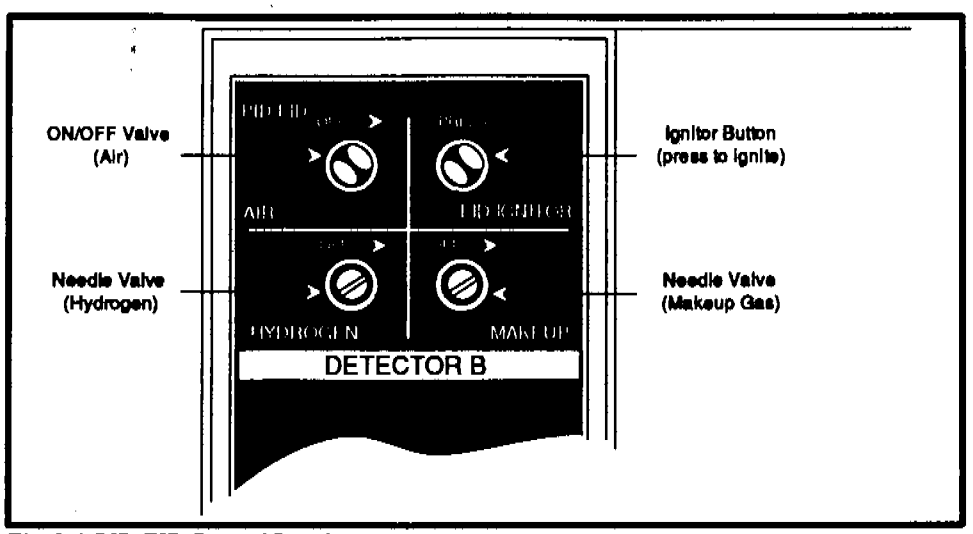


Fig. 2.4 PID/FID Control Panel

WARNING: *Never adjust the Gas Flow Module needle valves to the point of shut-off (fully clockwise).*

The Gas Flow Module provides two needle valves with ON/OFF control for hydrogen and makeup gases, one valve with ON/OFF control for air, and an ignition switch, as shown in Fig. 2.4. The needle valve adjustment is located inside the valve knob. The valve knob provides the ON/OFF function so that settings are retained.

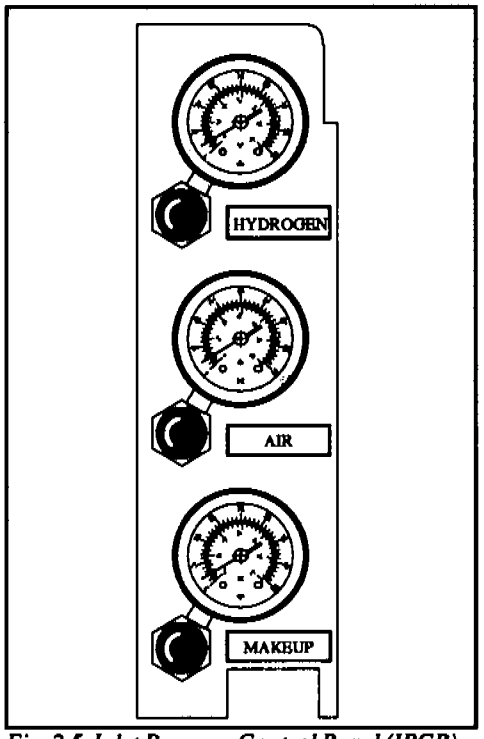


Fig. 2.5 Inlet Pressure Control Panel (IPCP)

OI Analytical offers the **Inlet Pressure Control Panel (IPCP)** gas regulator (OI Analytical #204776) as an accessory that improves performance and facilitates operation (see Fig. 2.5). This option is strongly recommended for all applications. Without the IPCP, pressure changes in the gas supply source may significantly change flow rates in the system, which in turn will change detector response. The IPCP isolates gases from minor outside disturbances, such as opening or closing valves on other systems or temperature changes on gas cylinder regulators. A second function of the IPCP is to provide control for detectors with different line pressure requirements for a given gas.

Chapter 3
Installation

The following instructions apply to standard installations of 4410 FIDs on HP 5890A and HP 5890 Series II GCs. The FID is intended to be installed only in tandem with an OI Analytical 4430 PID. Some installations will be to retrofit a FID onto an existing 4430 PID or to install a 4450 PID/FID. These installations are considered "standard," while other applications are "non-standard." Non-standard installations require specific, and often unique, system knowledge that exceeds the scope of this manual. For non-standard installations, contact the OI Analytical Service Department at 1-800-336-1911.

For simplicity, the following procedures apply to a retrofit installation where a 4430 PID has already been installed. For installation of the PID detector, refer to the PID Operator's Manual. In either case, a common starting point exists.

The common starting point requires the following settings:

- All electrical equipment is unplugged.
- All gas pressures are **OFF**.
- Side, top, and rear panels of the GC are removed.
- The PID is installed in the "B" detector position or through a valve box.
- PID-electrometer connections are made to the "Detector B" circuit board in the GC.
- The "Detector A" electrometer circuit is available for the FID.
- The PID heater and temperature sensors are installed in the detector base and connected to the GC electronics.
- The PID exhaust jet assembly is not installed.

NOTE: The Tandem PID/FID should not be installed in the "A" detector position as this would physically prevent installation of other detectors in the "B" position.

FID/HP 5890 GC Installation

Install Gas Flow Module Output Lines

Each of the output gas lines from the Gas Flow Module is a 1/16" stainless steel tube with a button-like fitting on one end. These button fittings are installed in the bottom port (auxiliary makeup gas) and two side ports (hydrogen and air) of the Module. Each of the three tubes is bent flat for shipping but, when appropriately twisted, fit neatly into the shape needed for installation. The tube lengths supplied are appropriate for installation in the "Detector B" position. The tubes may be cut to length and deburred for installation in other locations. The tubes (especially their cut ends) must remain free from flow restrictions and from contamination that may interfere with analysis. Install the output gas lines according to the following procedure:

- Connect a 1/16" stainless steel gas line (OI Analytical #188474) to the auxiliary makeup output port at the bottom of the Gas Flow Module. Place a manifold O-ring (OI Analytical #185116) from the Gas Flow Module Start-up Kit into the output port, pressing the button fitting against the manifold O-ring (see Fig. 3.1, page 11).

- Secure the fitting with the manifold plate (OI Analytical #197772) with the plate screw that is provided. The holes in the manifold plate are not symmetric: only one orientation is appropriate.

- Connect the other two 1/16" stainless steel gas lines to the hydrogen and air output ports at the side of the Gas Flow Module. Install these gas lines in a manner similar to the auxiliary makeup line (see Fig. 3.1, page 11).

- The free end of the stainless steel gas lines will be installed on the detector later.

Install Gas Flow Module

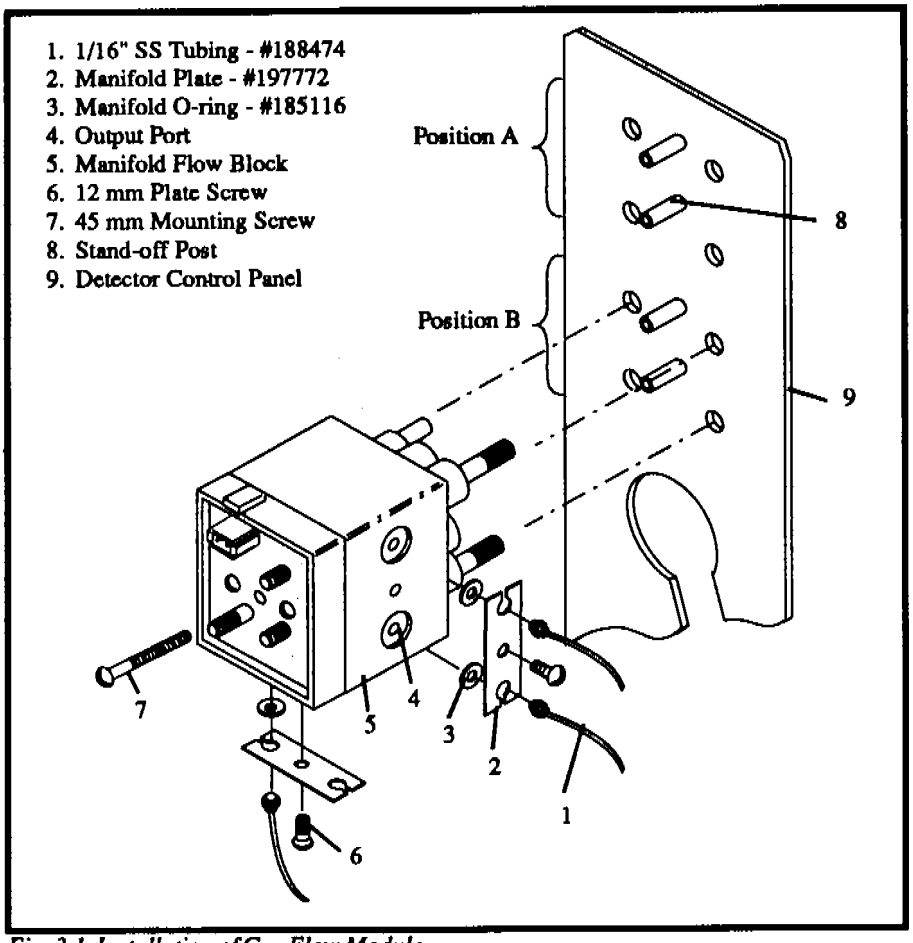


Fig. 3.1 Installation of Gas Flow Module

- Slip the Module into place on the backside of the front panel as shown in Fig. 3.1. The Module fits over the two stand-off posts and is fastened with the M4 x 45 mm mounting screw in the center location ***(do not over-tighten the screw)***. Check the ON/OFF valves for freedom of movement. If the valve knobs are difficult to adjust, loosen the mounting screw and adjust the ON/OFF knob position until approximately half-open. Re-tighten the mounting screw and again check for valve knob freedom of movement.

- Remove the plastic protective backing to expose the adhesive on the PID/FID plastic face plate (OI Analytical #192211), slip it over the valve knobs on the front panel of the GC, and press it firmly in place.

Install Optional IPCP

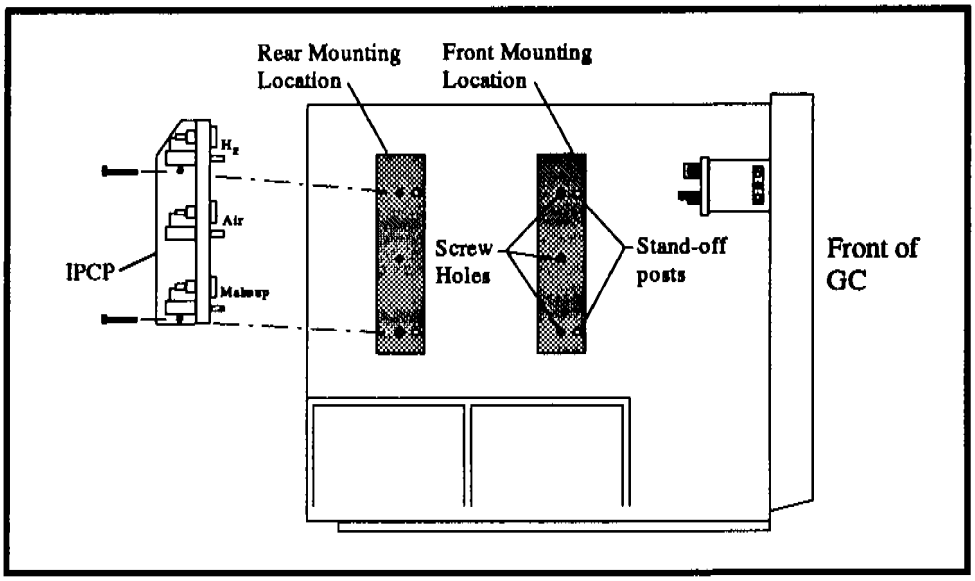


Fig. 3.2 IPCP Installation

Install the IPCP according to the following procedure:

- Turn OFF the GC's main power switch and disconnect the instrument from line power.
- Shut OFF supply gases at their main sources.
- Remove the left side panel of the GC by first removing the two screws near the bottom of the panel then sliding the panel toward the rear of the GC and lifting it off.
- Locate the two sets of stand-off posts on the left side of the GC, as shown in Fig. 3.2. The front location is used if only one IPCP is installed. Both the front and rear locations are used if two IPCPs are installed.
- Orient the IPCP so that the front stand-off posts project through the two larger holes in the IPCP. Fasten the panel to the GC with the two screws provided.

WARNING: *Do not apply supply line pressure to the regulators until it is time to set gas flow for the PID/FID.*

- Disconnect supply gas lines from the flow controls on the main flow panel and reconnect them to the IN fittings on the appropriate IPCP pressure regulator. The IPCP regulators receive 1/8" gas lines from hydrogen, auxiliary makeup gas, and air sources.
- Disconnect one line at a time and connect it to the IPCP before proceeding with the next gas line. The IPCP fittings require a 7/16" open-end wrench. The Swagelok fittings should be finger-tight plus approximately 3/4 of a turn more (use two open-end wrenches). If two Gas Flow Modules are installed on the GC and they require different supply pressures for a given gas, the higher pressure requirement is plumbed using a tee fitting prior to the input of the IPCP regulator. The lower pressure requirement is provided through the regulator of the IPCP. Run 1/8" gas lines from the tee fitting to the higher pressure Gas Flow Module requirement.
- Apply the appropriate self-adhesive gas supply labels to the front of the IPCP control panel.

Install Gas Flow Module Input Lines

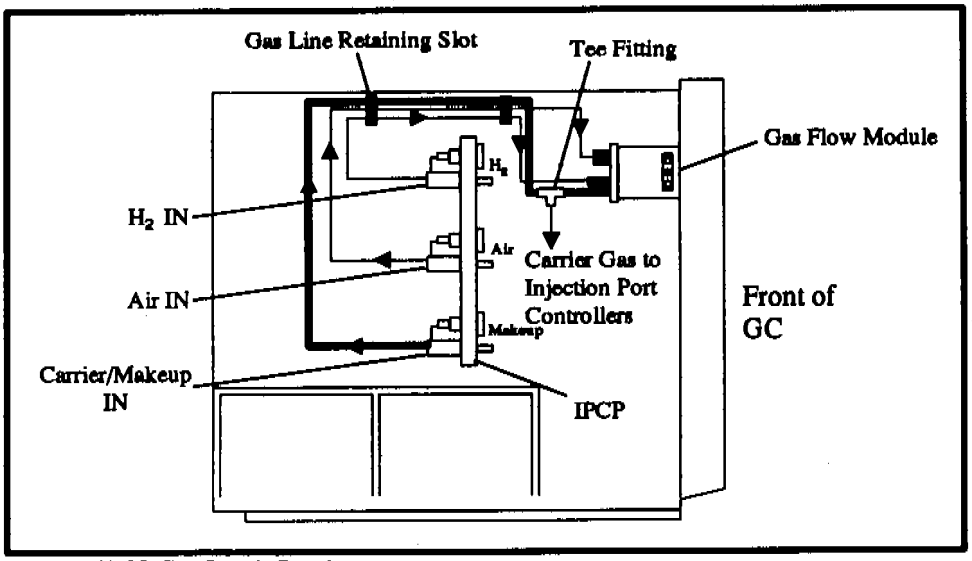


Fig. 3.3 IPCP Gas Supply Routing

- If an IPCP is installed, run 1/8" gas lines from the IPCP OUT fittings to the inlet fittings of the Gas Flow Module using a 1/8" brass ferrule set (OI Analytical #196089) (provided in the FID Start-up Kit), a 1/8" hex tube nut (OI Analytical #128108) (provided in the FID Start-up Kit), and a piece of 1/8" copper tubing (provided in the IPCP Start-up Kit). All Swagelok compression fittings should be finger-tight plus approximately 3/4 of a turn more (use two open-end wrenches). The side of the auxiliary makeup line will need to be split with a tee or cross fitting to supply gas to the injection port controllers, as shown in Fig. 3.3.

- If an IPCP is not installed, connect 1/8" supply lines to the inlet fittings of the Gas Flow Module using a 1/8" brass ferrule set (OI Analytical #196089) and a 1/8" hex tube nut (OI Analytical #128108) provided in the FID Start-up Kit. All Swagelok compression fittings should be finger-tight plus approximately 3/4 of a turn more (use two open-end wrenches).

Install 4410 FID

The following instructions assume that the 4430 PID is correctly installed on the GC. For PID installation, see Chapter 3 of the 4430 PID Operator's Manual.

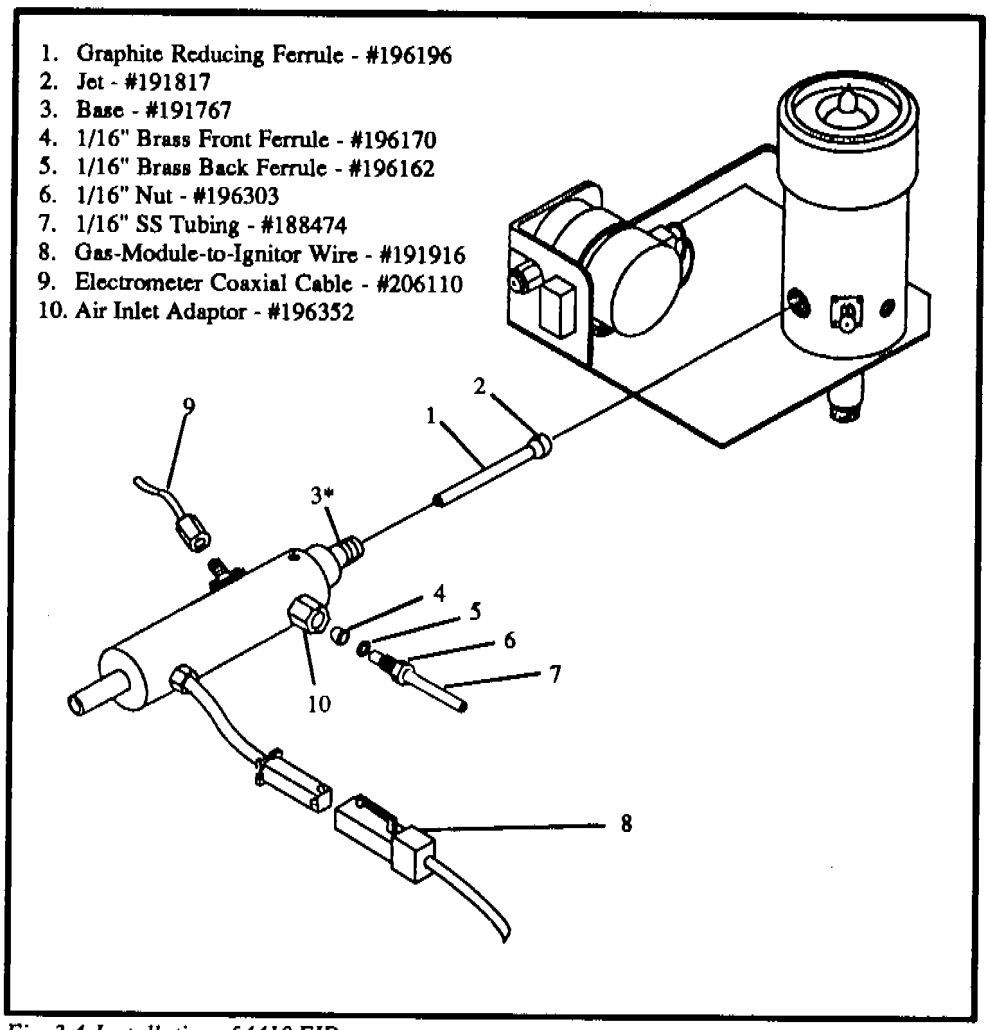


Fig. 3.4 Installation of 4410 FID

WARNING: *Do not use graphite/ Vespel® composite ferrules for this assembly as they do not provide an adequate seal.*

CAUTION: *The set screw holds the FID body in place and does not exert any sealing force on the O-ring(s). Do not over-tighten. Tighten the set screw using the thumb and index finger on the shaft of a 0.050" Allen wrench.*

- Remove the FID base from the body by loosening the Allen set screw using a 0.050" Allen wrench.
- Slide a graphite reducing ferrule onto the jet.
- Screw the FID base (with the two installed O-rings*) and the jet (with the graphite reducing ferrule) into the exhaust port of the PID, as indicated in Fig. 3.4. Make certain the jet is firmly seated against the recess in the PID exhaust port. Tighten the base with a 5/16" wrench.
- Attach the FID body to the base of the FID by tightening the Allen set screw. Gently check the tightness of the set screw.

* Base designs prior to December 1991 require one O-ring.

Install Detector Input Gas Lines

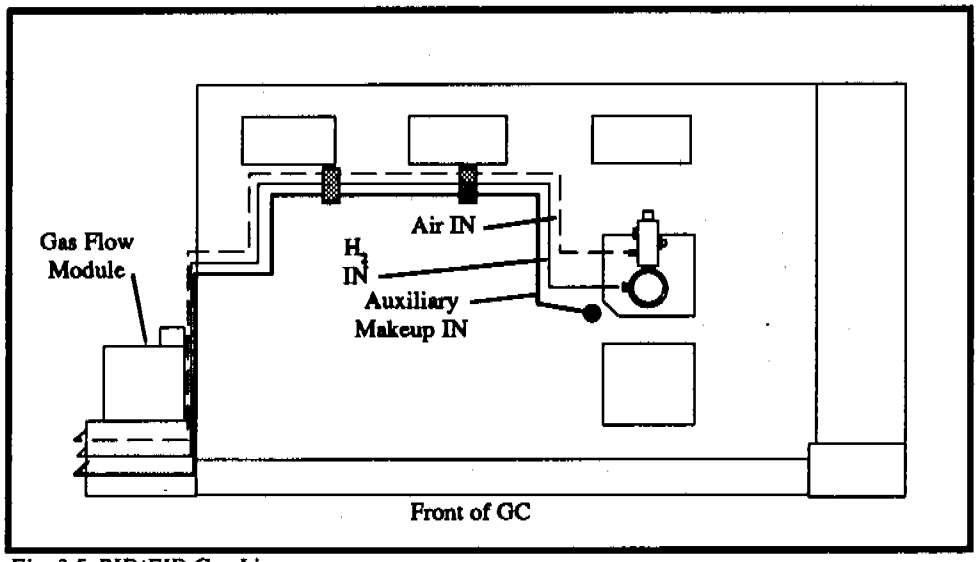


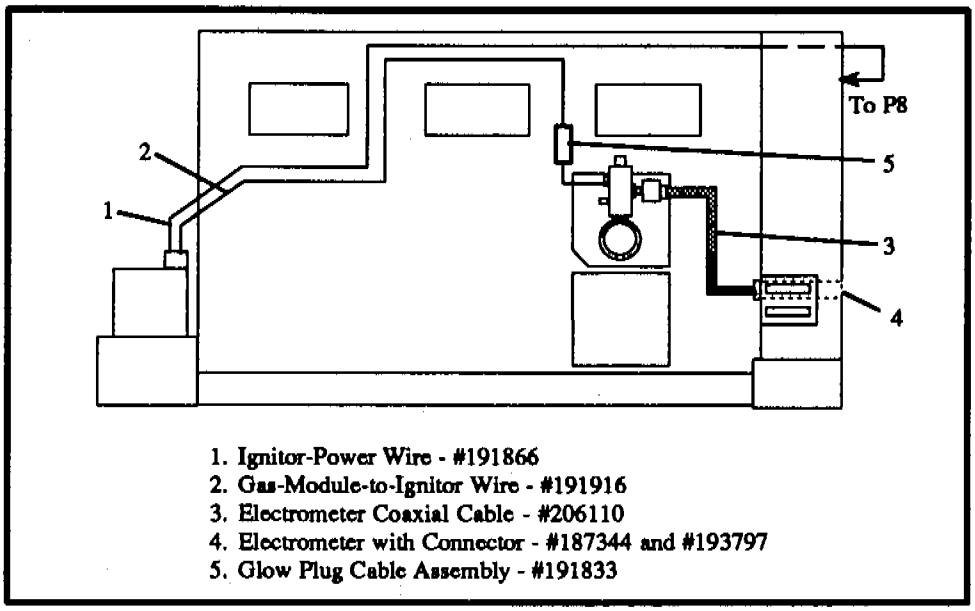
Fig. 3.5 PID/FID Gas Lines

- The hydrogen and auxiliary makeup lines are terminated and connected as directed in the PID Operator's Manual (Chapter 3, Installation on HP 5890). Route these gas lines as shown in Fig. 3.5.

- Route the air line according to Fig. 3.5. Connect the open end to the air inlet adapter on the FID body using a 1/16" nut (OI Analytical #196303) and a 1/16" brass ferrule set (OI Analytical #196162 and #196170) provided in the FID Start-up Kit, as shown in Fig. 3.4, page 14.

Install Ignitor Wires

WARNING: *The GC electrical components contain high voltage. Turn OFF the GC and the detector power and disconnect all line power.*



1. Ignitor-Power Wire - #191866
2. Gas-Module-to-Ignitor Wire - #191916
3. Electrometer Coaxial Cable - #206110
4. Electrometer with Connector - #187344 and #193797
5. Glow Plug Cable Assembly - #191833

Fig. 3.6 FID Electrical Connections

WARNING: *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.*

- Connect the gas-module-to-ignitor wire (OI Analytical #191916) from the connector on the pigtail of the glow plug assembly to the inboard terminal of the ignition switch on the Gas Flow Module, referring to Fig. 3.6.

- Connect the ignitor-power wire (OI Analytical #191866) to the outboard terminal of the ignition switch. Run this wire through the wire cable tray along the back of the GC to P8 (HP 5890 Series II) or J8 (HP 5890A) on the GC motherboard (see Fig. 3.7).

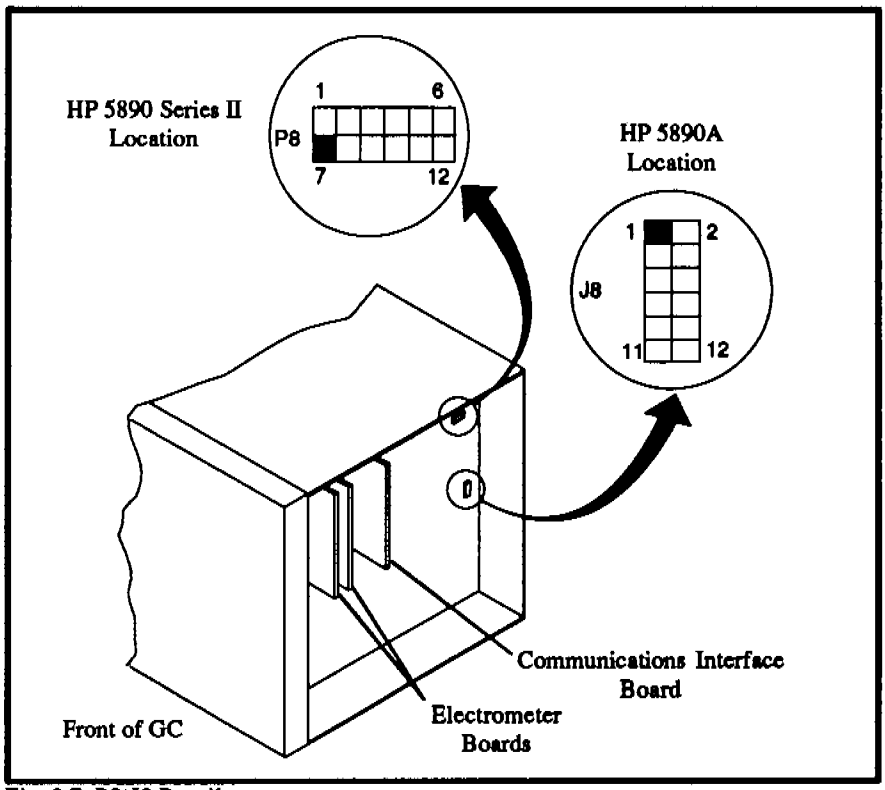


Fig. 3.7 P8/J8 Detail

- Carefully disconnect the P8 or J8 plug and insert the ignitor-power wire terminal into position 7 on P8 or position 1 on J8, as shown in Fig. 3.7. Pull on the plug itself rather than on its wires to prevent breakage. Check for positive latching. Replace the plug in its socket on the motherboard.

Install Electrometer Board

WARNING: The GC electrical components contain high voltage. Turn the GC and the detector power OFF and disconnect all line power.

WARNING: 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.

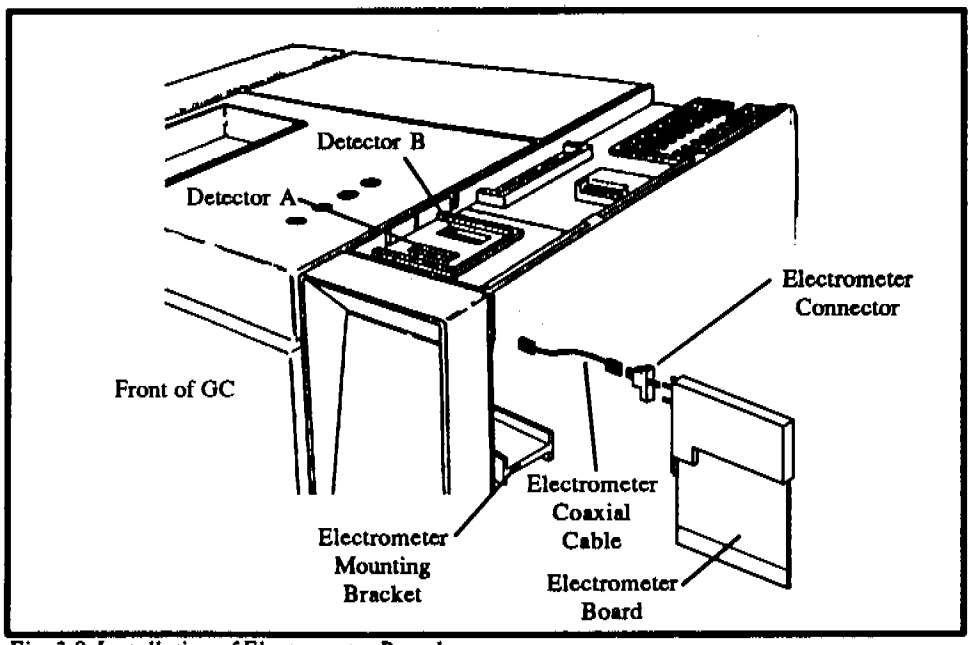


Fig. 3.8 Installation of Electrometer Board

The following instructions apply to an FID HP electrometer board.

- Attach the electrometer connector (OI Analytical #193797) to the HP electrometer board (OI Analytical #187344), referring to Fig. 3.8. Use the three nuts and washers provided with the electrometer board.
- Attach the electrometer coaxial cable (OI Analytical #206110) to the electrometer connector.
- Feed the free end of the electrometer coaxial cable through the round hole in the "Detector A" area of the GC wall.
- Push the electrometer board into the "A" slot while gently pulling the electrometer coaxial cable through the hole.
- Connect the free end of the electrometer coaxial cable to the coaxial connector on the FID body. Tighten the coaxial connector nut approximately 1/4-turn past finger-tight. ***Do not over-tighten.***

Set Gas Pressure with the IPCP

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. Do not turn on the hydrogen without a column or cap attached to the detector inlet.*

- Turn all three black ON/OFF valve knobs on the Gas Flow Module clockwise until a mechanical stop is felt. This closes the hydrogen, auxiliary makeup gas, and air valves.
- Pressurize the hydrogen, auxiliary makeup gas, and air lines at the supply line source to approximately 50-60 psi. If the gas lines are split prior to the IPCP for Gas Flow Modules requiring dissimilar pressures, set the supply line source to the higher pressure requirement. The Gas Flow Module with the lower pressure requirement will be supplied and controlled by the IPCP.
- Adjust the three IPCP regulators to approximately 40-50 psi.
- Check all gas connections for leaks.
- Attach a flowmeter to the exhaust port of the FID. Confirm the PID vent valve is in a closed position.
- Set the column flow rate according to the column manufacturer's instructions and the intended analytical method.

Table 1 Gas Flow Rates

Gas	Individual	Additive
Carrier + Makeup	30 ± 2 ml/min	30 ml/min
Hydrogen (PID Sweep Gas)	35 ± 2 ml/min	65 ml/min
Air	165 ± 15 ml/min	230 ml/min

The PID sweep gas is used as the FID hydrogen gas supply. Therefore, changing the FID hydrogen flow changes the PID sweep gas rate.

NOTE: The gas flow rates recommended should provide reliable FID performance. However, optimal performance for any chromatographic system may require minor flow rate adjustments to the flow rates given in Table 1. Increasing the hydrogen flow rate gains flame stability at the expense of sensitivity. Increasing air flow rate gains sensitivity at the expense of flame stability.

- Set the auxiliary makeup gas flow rate according to Table 1, compensating for the additive column flow rate. First open the ON/OFF valve and then adjust (with a small screwdriver) the needle valve located in the center of the black ON/OFF valve knob. The needle valve opens counterclockwise.

  NOTE: When opening the ON/OFF valves to measure gas flows, allow air to be purged from the gas lines to ensure that the correct flow is measured.

- Set the hydrogen flow rate in a manner similar to the auxiliary makeup gas. Refer to Table 1.
- Set the air flow rate by first opening the ON/OFF valve and then adjusting the pressure at the IPCP until the desired flow rate is achieved (see Table 1). The air flow rate can be set only by adjusting the IPCP regulator pressure: there is no needle valve for controlling air flow.

- Disconnect the flowmeter from the FID exhaust port.
- Re-check all gas connections for leaks.
- When all fittings are known to be leak-free, replace the side panel on the GC.

Set Gas Pressure without the IPCP

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. Do not turn on the hydrogen without a column or cap attached to the detector inlet.

- Turn all three black ON/OFF valve knobs on the gas flow module clockwise until a mechanical stop is felt. This closes the hydrogen, auxiliary makeup gas, and air valves.
- Pressurize the hydrogen, auxiliary makeup gas, and the air lines at the supply line source to approximately 50-60 psi.
- Check all gas connections for leaks
- Attach a flowmeter to the exhaust port of the FID. Confirm the PID vent valve is in a closed position.
- Set the column flow rate according to the column manufacturer's instructions and the intended analytical method.

Table 1 Gas Flow Rates

Gas	Individual	Additive
Carrier + Makeup	30 ± 2 ml/min	30 ml/min
Hydrogen (PID Sweep Gas)	35 ± 2 ml/min	65 ml/min
Air	165 ± 15 ml/min	230 ml/min

The PID sweep gas is used as the FID hydrogen gas supply. Therefore, changing the FID hydrogen flow changes the PID sweep gas rate.

NOTE: The gas flow rates recommended provide reliable FID performance. However, optimal performance for any chromatographic system may require minor flow rate adjustments to the flow rates given in Table 1. Increasing the hydrogen flow rate gains flame stability at the expense of sensitivity. Increasing the air flow rate gains sensitivity at the expense of flame stability.

- Set the auxiliary makeup gas flow rate according to Table 1, compensating for the additive column flow rate. First open the ON/OFF valve and then adjust (with a small screwdriver) the needle valve located in the center of the black ON/OFF valve knob. The needle valve opens counterclockwise.

  NOTE: When opening the ON/OFF valves to measure gas flows, allow air to be purged from the gas lines to ensure that the correct flow is measured.

- Set the hydrogen flow rate in a manner similar to the auxiliary makeup gas. Refer to Table 1.

- Set the air flow rate by first opening the ON/OFF valve and then adjusting the regulator at the supply pressure until the desired flow rate is achieved (see Table 1). The air flow rate can be set only by adjusting the supply pressure: there is no needle valve for controlling air flow.
- Disconnect the flowmeter from the FID exhaust port.
- Re-check all gas connections for leaks.
- When all fittings are known to be leak-free, replace the side panel on the GC.

Chapter 4
Operation

In the last chapter, installation of the 4410 FID was described. This chapter deals with the operation of the detector. These instructions assume that the procedures outlined in Chapter 3 have been completed. Because the 4410 FID is designed to be operated only in tandem with the OI Analytical 4430 PID, operation of the FID is dependent on an understanding of the PID. For this reason, it is recommended that the operator review the operation of the PID. Of particular importance is an understanding of the following:

- 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 supplied by the GC.

Each of these aspects of detector operation is covered in the GC or the PID Operator's Manual.

General Start-up Settings

Detector Base Temperature:	Determined by PID requirements, 200° C or 20° C above highest column temperature (270° C maximum)
Range Setting on GC:	2^6
Attenuation on GC:	2° (does not affect output used)
Zero Offset on GC:	0.0
Signal Output Scale:	0-1 Volt full scale, or as required by data system
FID Gas Flows:	H_2 — 35 ± 2 ml/min Air — 165 ± 15 ml/min He — Total of column and makeup = 30 ± 2 ml/min

General Operation

It is assumed the FID and column are properly installed, gases are set properly, the PID is operating correctly, and the system is free of leaks. Refer to Chapter 3 for setting gas flows.

- Turn ON the GC's main power and follow applicable instructions.
- Set the GC's setpoints for intended analytical method.
- Open the three ON/OFF valves for hydrogen, air, and auxiliary makeup gas.

CAUTION:
The PID and the FID may be hot.

- Ignite the FID by pressing the ignition button on the Gas Flow Module. Ignition is indicated by an audible "pop" and by visible condensation on a small, cool mirror or brightly polished wrench held temporarily near the exhaust port of the FID. ***Do not lean over the detector to see if it is ignited.***

NOTE: If using helium as the makeup and carrier gas, igniting the detector may be facilitated by turning OFF or venting the helium flows. Reopen the helium gas ON/OFF valves (or close the PID vent valve) once the flame lights.

Expected Signal Dynamics at Start-up

When the hydrogen flame is ignited in a new FID, the output signal typically becomes saturated, then it decays to a value between 10 and 20 picoamps within 10 hours. This delay is due to the slight contamination occurring during assembly and installation. After a new FID has been operated continuously for several days, a baseline signal of 5-10 picoamps is expected for clean systems using the properly conditioned commercial chromatographic grade gases suggested in this manual. A persistent steady high background current generally indicates impurities in the gas delivery system.

Chapter 5
Maintenance

This chapter discusses the maintenance of the 4410 FID. Due to the simple design of the 4410 FID, little maintenance is required.

The jet, the collector electrode, and the FID body may need to be cleaned or replaced due to corrosion or chemical deposits, depending on frequency of use, type of sample and solvent used, column type, and gas purity. If a contaminated detector is suspect:

CAUTION:
The PID and the FID may be hot.

- Turn OFF the gas flows to the detector and shut OFF the GC power.
- Allow the detector to cool down several minutes.
- Remove the short coaxial cable from the electrometer connector assembly and the 1/16" nut and ferrule at the air inlet fitting.
- Disconnect the ignition wire at the pigtail of the glow plug assembly.
- Remove the FID base by unscrewing the base from the PID exhaust port using a 5/16" open-end wrench. When standing in front of the GC, the base will unscrew in a clockwise rotation.
- Loosen the 0.050" Allen set screw from the FID body. Remove the FID body by gently pulling it away from base. ***The set screw should be loosened fully before removal of the FID body to avoid O-ring damage.***
- Visually inspect the interior of the FID body, the jet, the collector electrode, and the two brown Vespel® ferrules. If necessary, clean or replace contaminated parts according to Table 2 on page 24.

WARNING: Methanol is flammable and toxic. See the Materials Safety Data Sheet for methanol and use appropriate caution.

WARNING: The internal components of the FID may be hot.

Table 2

Symptom	Contamination	Cause	Solution
Black deposits	Carbon soot	High concentration of solvent and analyte	Scrub interior of collector electrode with a cleaning brush, then rinse with methanol.
White coloration	Silica particles	Combustion of column stationary bleed	Remove the glow plug cable assembly from the FID body. Clean with methanol, being careful not to damage the electrometer connector assembly.
Dark orange deposits	Surface oxidation (rust)	Excessive water entering the detector or production from an air rich flame	Place the FID body, jet, collector electrode, and the two Vespel® ferrules in an ultrasonic bath of methanol for approximately 30 min. Dry all parts in a suitable oven at 40° C for 15-20 min. Replace any contaminated or difficult to clean components.
Green to blue-green coloration or corrosion	Acid formation	Combustion of analytes and solvents, especially chlorinated compounds in the presence of excessive water	Replace defective parts. Check the hydrogen and air gas flows, adjust if necessary.

CAUTION: The Allen set screw holds the body in place and does not exert any sealing force on the O-ring. Do not over-tighten. Tighten the set screw using thumb and index finger on the shaft of a 0.050" Allen wrench.

- Inspect the two viton O-rings* attached to the FID base and replace if discolored, chalky, cracked, and/or brittle.
- Remove the graphite reducing ferrule from the PID exhaust port. Remove any remaining graphite particles with compressed air or other suitable means. ***Graphite particles in the PID tower and in the sample path to the FID may significantly affect the PID and FID performance.***
- Slide the jet into a new graphite reducing ferrule and screw the FID base (with the jet and ferrule) firmly into the exhaust port of the PID.
- If necessary, reconnect the glow plug assembly to the FID body.
- Reassemble the FID by sliding the detector body firmly onto the base and gently tightening the Allen set screw. ***Do not over-tighten.***

* FID base designs prior to December 1991 require one O-ring.

- Reconnect the ignitor glow plug assembly wires, the stainless steel air line, and the electrometer coaxial cable.
- Turn ON the power to the GC and open all gas supplies (check for leaks and reconfirm gas flow rates).
- Ignite the detector, allowing it to stabilize while monitoring the baseline signal to confirm that it decays to a value between 10 and 20 picoamps in approximately 10 hours. After the FID has been operating continuously for several days, a baseline signal of 5 to 10 picoamps should be expected for a properly cleaned system using the required grade gases and gas traps.

NOTES

Chapter 6
Troubleshooting

Once the 4410 FID is installed and operating properly, make note of the signal display and a typical baseline on the recorder or data system with no zero offset. A typical chromatogram for comparing response is also useful. Care should be taken to examine the entire GC system in any troubleshooting attempt.

Generally, there are three causes of FID signal malfunctions: (1) contamination, (2) errors in setting gas flow rates, and (3) electrical problems. Refer to the Troubleshooting Chart at the end of this chapter for an overview of possible symptoms and their corresponding solutions. Each listed symptom may be caused by more than one potential problem. Before using this guide, please become thoroughly familiar with the operation and maintenance information in the previous two chapters.

Contamination

Low quality gas will lead to system contamination. The purity of the hydrogen, air, and auxiliary makeup gases is often the limiting factor in FID signal quality. The effect of gas purity will vary according to method, column type, stationary phase chemistry, sample concentration, injection technique, effectiveness of gas line filters, etc. In general, the commercial grade gases suggested in this manual with appropriate conditioning will suffice.

Inappropriate sample preparation can lead to system contamination in a variety of ways. If the sample contains material that will not readily and completely elute from the system, contamination is likely. If the sample corrodes or degrades any part of the system, contamination will also occur. Replace and/or clean the affected parts.

The surfaces of the gas system must be analytically clean at the time of final assembly of the system. Unintended influx of any gas through leaks or permeable components must be avoided. Leaks are most easily detected by charging the system with helium and then searching the system's exterior with a helium leak detector. Do not use permeable materials in the system. Transpermeation or out-gasing from these materials will raise the background signal and sometimes yield false or misleading peaks.

Gas Flow Rates

Errors in measuring and setting gas flow rates for hydrogen, air, and auxiliary makeup gas will cause system problems. Difficulty in igniting the FID may be caused by setting the gas flow rate outside the practical flammability envelope. A flame that is hydrogen-lean or air-rich will be difficult to ignite. If the flame is hydrogen-rich, it is probable that deposits of sample residue on the jet and collector will occur and cause reduced sensitivity.

Electrical Problems

FID electrical problems can be divided into two categories: ignitor difficulties and signal problems. Ignitor difficulties are usually due to a defective glow plug assembly, ignitor switch, ignitor cables, or improper ignitor cable connections. Refer to Chapter 3 for the proper ignitor cable installation. Check all connections and, if necessary, identify defective component(s) in a logical, systematic fashion. FID signal problems are usually due to defective electrical components or incorrect signal pathways.

Verify the signal pathway with the aid of the diagnostic chromatogram test built into the HP GC. A perfect signal plot indicates a correct pathway. The problem, such as a lost or noisy signal, will then be chromatographic (i.e., a contaminated detector, leaks, etc.). Noisy peaks on the plot indicate noise in the electrical system usually due to defective parts or poor electrical connections. No peaks on the test chromatogram indicate improper electrical connections, improper signal assignment, or defective components. To isolate electrical component problems, use a modular approach.

Overview

Use the available technical resources and a knowledge of the analytical system to aid in diagnosing signal problems. Because the GC analytical system is modular, use a process-of-elimination approach to identify and eliminate causes of problems. Use the instruction manuals of the various system components to assist in solving unusual or particularly difficult problems.

FID Troubleshooting Chart

Symptom	Probable Cause	Corrective Action
noise on baseline	loose electrical connection	tighten connection
	vibration of cable	re-route cable
	contamination in detector	clean/replace contaminated parts
	leaks in system	identify and fix leaks
	improper ground connection	check ground continuity
	signal cable routed near power cable	re-route cable
	faulty electrical component	verify signal pathway and diagnose electrical system — replace components as necessary
spike (random)	particle contamination in FID flame	clean or replace jet and/or collector (solid build-up is caused by column bleed, improper sample preparation, injection volume being too large, or incorrect gas flow rates)
	contamination of FID components	
	loose or faulty electrical components	check connections and diagnose electrical system
high or increasing background in isothermal mode	electrical noise	verify signal pathway and diagnose electrical system in modular fashion — correct as necessary
	contaminated gas	use higher purity gases
		condition gases with filters and/or traps
	contaminated inlet system, column bleed, PID contamination, dirty jet, contaminated Gas Flow Module, and/or improper sample preparation	verify signal pathway and diagnose system in modular fashion — correct as necessary

Symptom (cont'd)	Probable Cause (cont'd)	Corrective Action (cont'd)
broad eluting peaks	contamination in system	diagnose system in modular fashion — correct as necessary
	large solvent peak interruption	decrease sample volume
	column overload	decrease sample volume
	restrictions in PID or FID	check all gas flow paths for obstructions, particles, and graphite
	improper carrier flow rate	adjust flow rate
flame goes out or will not light	incorrect gas flow rates	adjust gas flow rates (review operating conditions)
	gas leak	fix leaks
	improper ignitor electrical connections	diagnose ignitor electrical system in modular fashion — correct as necessary
	damaged or dirty jet	replace jet
	defective glow plug assembly	replace glow plug
	improper column or FID installation	correct as needed (review installation procedures)
	restriction in PID exhaust port	check installation of FID reducing ferrule, jet, and detector base
	large solvent peak interruption	reduce sample size
loud "pop" during flame ignition	incorrect gas flow rates (**NOTE:** a soft audible "pop" is normal if flame lights)	adjust gas flow rates (review operating conditions)
no response	faulty electrical components or connections	diagnose electrical system in modular fashion — correct as necessary
	excessive gas leaks	fix leaks

Symptom (cont'd)	Probable Cause (cont'd)	Corrective Action (cont'd)
no response	flame not lit	check flame (review operating conditions)
	improper FID installation	correct as needed (review installation procedures)
reduced sensitivity	incorrect gas flows	adjust gas flow rate
	contaminated detector	clean or replace contaminated components (review maintenance procedures)
	gas leaks	fix leaks
	electronic noise	verify signal pathway and diagnose electrical system in modular fashion — correct as necessary
	damaged jet	replace jet
	restriction in PID or FID	check all gas flow paths for obstructions, particles, and graphite

NOTES

Chapter 7
Replacement Parts

In Chapter 2, the various components of the 4410 FID were identified and described. This chapter is a listing of the order numbers for these components and for other replacement parts and support items. Replacement parts that OI Analytical considers expendable (EXPD) are marked as such. Expendable components are ones that are to be replaced regularly or are easily broken or deformed.

Part Name	Part #	U/M	EXPD
Base - FID	191767	ea	
Cable - Analog (O-1V)	185850	ea	
Cable - Coaxial Electrometer	206110	ea	
Cable - Gas Module to Ignitor	191916	ea	
Cable - Glow Plug Assembly	191833	ea	
Cable - Ignitor Power	191866	ea	
Casing - FID Body	191759	ea	
Connector - Electrometer	193797	ea	
Connector - Coaxial Cable	191791	ea	
Electrode - Collector	191825	ea	
Ferrule - BR 1/16 Tube Back (5/pk)	196162	pk	*
Ferrule - BR 1/16 Tube Front (5/pk)	196170	pk	*
Ferrule - BR 1/8 Tube Set (10/pk)	196089	pk	*
Ferrule - GRP 1/8 x 1/16 Tube (5/pk)	196196	pk	*
Ferrule - Upper FID Body	191775	ea	
Ferrule - Lower FID Body	191783	ea	
Fitting - Adapter BR/NI 1/16 Tube (5/pk)	196352	pk	*
Fitting - BR 1/8 Cross Tube Male	186239	ea	
Fitting - BR 1/8 Female	128108	ea	
Fitting - BR 1/8 Port Tube	117721	ea	
Fitting - BR 1/8 Tee Tube Male	124750	ea	
Fitting - BR/NI 1/16 Nut Male (5/pk)	196303	pk	*
Jet - 0.3 mm I.D.	191817	ea	*
Kit - Gas Module PID/FID	192013	ea	
O-Ring VTN Base (5/pk)	210278	pk	*
O-Ring VTN Manifold (12/pk)	185116	pk	*
Plate - Manifold Block	197772	ea	
Screw - Allen Set 6-32 x 1/8	131961	ea	
Screw - Machine 2-56 x 3/16	144361	ea	
Standard - Detector 100 ppm	222919	ea	*
Sub Assembly - FID	191742	ea	
Tube - CU 1/8 x 0.070 I.D.	111427	ft	*
Tube - SS Gas Module Output	188474	ea	
Valve - Needle Gas Module	185587	ea	
Valve - ON/OFF Gas Module	191973	ea	

NOTES