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5383 PFPD
Pulsed Flame Photometric Detector
Operators Manual

USER MANUAL
4231-01

O·I·Analytical®

Notice

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Chapter 1 Introduction

OI Analytical's Model 5383 Pulsed Flame Photometric Detector (PFPD) represents the latest advance in flame photometric detector design, optimizing selective detection of sulfur, phosphorus, and 26 other compounds.

Detector Design

The 5383 PFPD consists of four principal components:

- 5383 detector assembly,
- 5383 detector electronics controller,
- 5383 detector pneumatics controller,
- Detector gas flow components,
- PulseView™ Monitor software.

The 5383 Detector Assembly consists of a base, body, combustor cap, ignitor assembly, light pipe, optical filter, photomultiplier tube (PMT) housing, PMT, heater, and temperature sensor. The 5383 detector electronics controller houses the signal processing and power supply electronics. The 5383 detector pneumatics controller houses the needle valve and flow control modules. The detector gas flow controls are made up of three gas flow controllers, gas mixing assemblies, and a precision needle valve. For models with manual flow controls, all components are located inside the detector pneumatics controller. For EPC-ready models, gas flow is controlled by the GC, and the mixing assemblies and needle valve are located inside the detector pneumatics controller.

Operating Principles

The PFPD uses a hydrogen and air mixture at a flow rate that does not support continuous combustion. The combustor is filled with an ignitable gas mixture, the flame is ignited, the flame propagates through the combustor, and it burns out when all the fuel is consumed. The cycle is repeated continuously at a rate of 3–4 hertz. The gas phase reactions produced by the propagating flame result in light emissions with specific luminescent spectra and lifetimes. The differences in specific emission lifetimes combined with the kinetics of the propagating flame allow both time and wavelength information to be used to improve the PFPD's selectivity and to decrease the observed noise, enhancing sensitivity. The propagating flame uses low combustible gas flow rates, increasing the relative analyte concentration. This is especially important for species such as sulfur that form dimers. In addition, using gated electronics permit the acquisition of two simultaneous, mutually selective chromatograms and permits rejection of noise occurring outside the specified gate window, further improving the PFPD's detectivity (Figure 1.1).

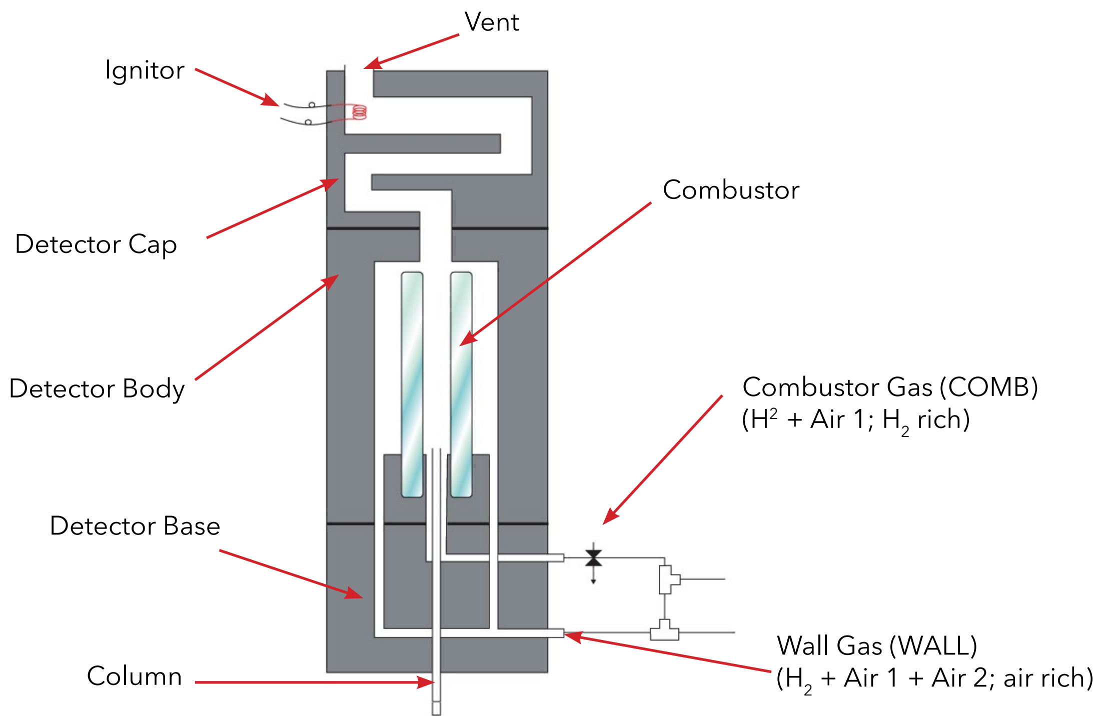


Figure 1.1. Combustor and Wall Gas Pathways

To retain all the emitting species within the combustor for increased sensitivity, a separate secondary flow of air and hydrogen (H_2) is continuously directed around the outside of the combustor. This flow fills the igniter cap with a fresh combustible gas mixture enriched with air for easier ignition. The primary and secondary gas flows are known as combustor gas (COMB) and wall gas (WALL), respectively. The pathways of these two gas mixtures within the detector are shown schematically in Figure 1.2.

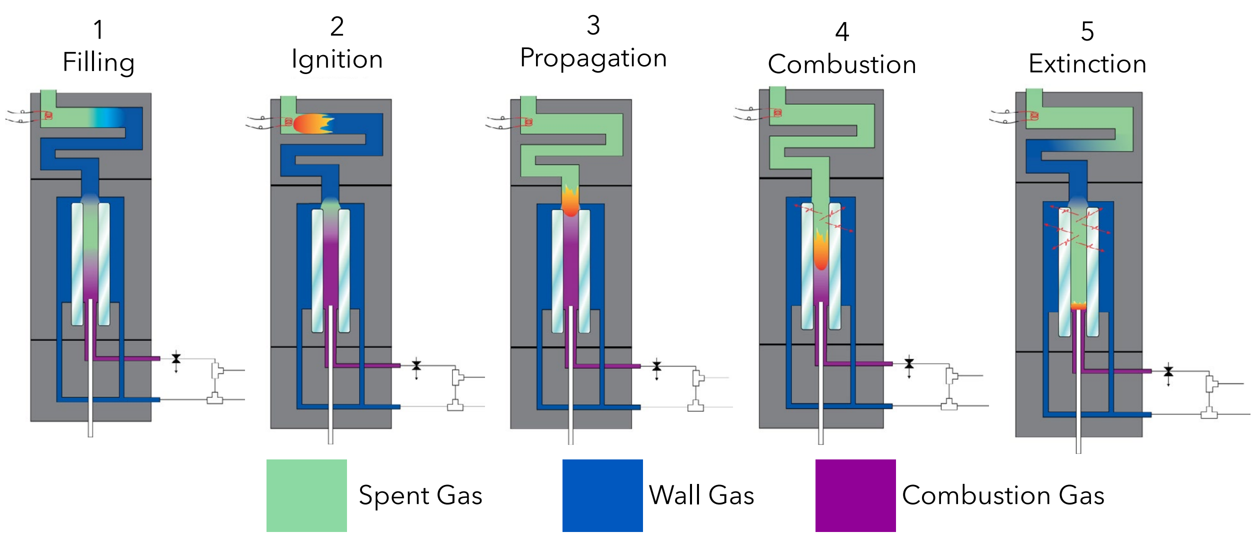


Figure 1.2. Flame Propagating Cycle

The pulsed flame propagation consists of the following steps (Figure 1.2):

Step 1: Filling	Detector cap pathway is filled with the air-rich combustible gas mixture of H_2 and air.
Step 2: Ignition	The gases are ignited by the hot filament of the ignitor.
Step 3: Propagation	The flame propagates downwards through the cap and the combustor, consuming the gas mixture.
Step 4: Combustion	Analytes are combusted within the flame to form the emitting species. The time-dependent emissions are directed down a light pipe, through an optical filter (not shown), and are detected by the PMT.
Step 5: Extinction	The flame is extinguished when it reaches the bottom of the combustor.

OI Analytical designed the PFPD's gas flow control system to allow the operator to regulate both the flow rates and the H_2:Air ratio of the combustor and wall gases. Figure 1.3 provides a flow diagram of the gas flow control and mixing system. Three manual or electronic gas flow controllers adjust the H_2 and air flow rates in the combustor gas (COMB) and wall gas (WALL) mixtures. The H_2 and Air 1 controls determine the gas mixture composition supplied to the combustor. The Air 2 regulator controls the flow rate through the WALL line and the ignition frequency. A fine-adjust needle valve controls the relative amounts of the gas mixtures that flow to the COMB and WALL lines.

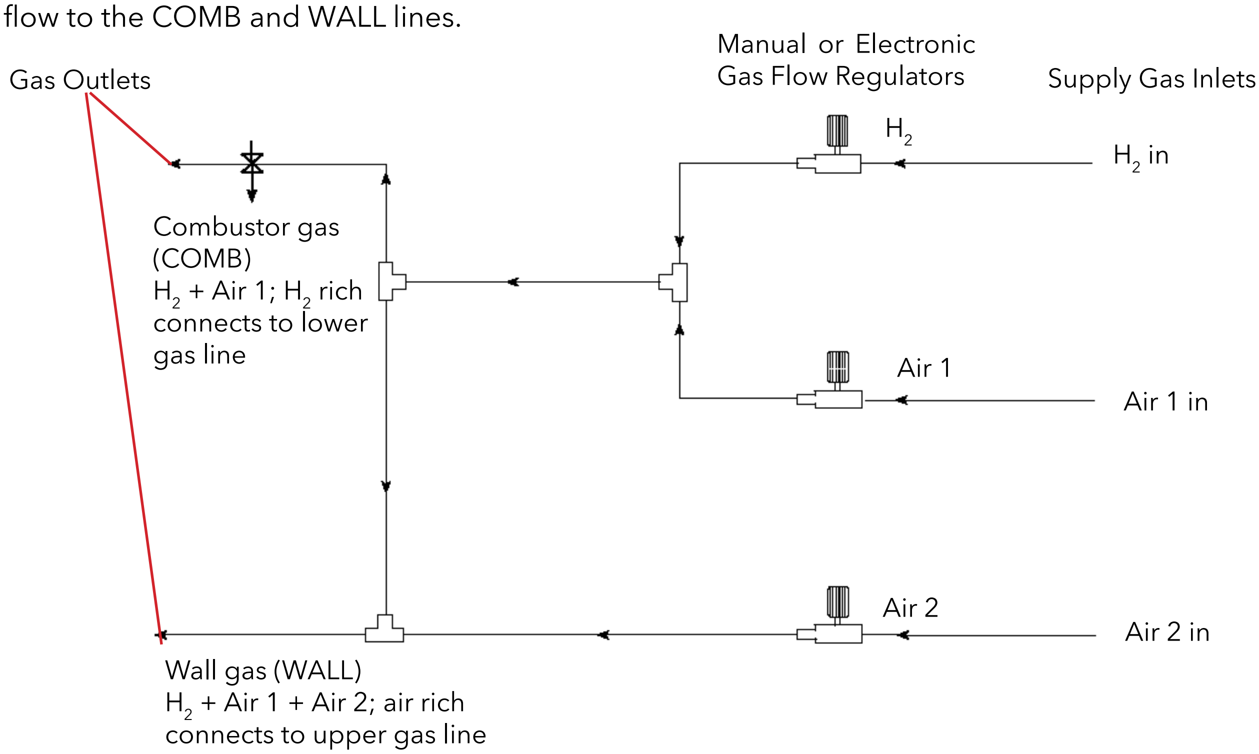


Figure 1.3. 5383 PFPD Diagram of Detector Gas Flow Control and Mixing System

The operator can easily manipulate the kinetics of the propagating flame and the associated lifetime of the chemiluminescence resulting from combustion by adjusting the gas flow ratios. (See Chapter 6, Operation, for a discussion of gas flow settings that optimize the PFPD's operating efficiency.)

The emission spectra detected by the PFPD are the product of the transmission of the emitted photons through the light pipe and filter and the spectral sensitivity of the photomultiplier tube. Operators can observe the element-specific emission profiles for species that are produced in the PFPD. With a conventional flame photometric detector (FPD), narrow-band pass interference filters minimize interference from carbon in the continuous flame. By contrast, the PFPD uses broadband-pass filters and time-based selectivity to minimize interference and increase optical throughput.

The background emission associated with the H_2-rich flame in the combustor consists of CH*, C_2*, and OH* species that exist in the flame. This emission is observed between approximately 1 and 3 msec and is due to the dynamics of the flame propagating through the combustor (Figure 1.4). The sulfur emission reaches its maximum between approximately 9 and 15 msec. The phosphorus emission reaches a maximum at approximately 3 to 4 msec and slowly decays, disappearing entirely by about 15 msec. Thus, sulfur and phosphorus have chemiluminescent lifetimes substantially different from one another, and longer than the background analyte-free emission.

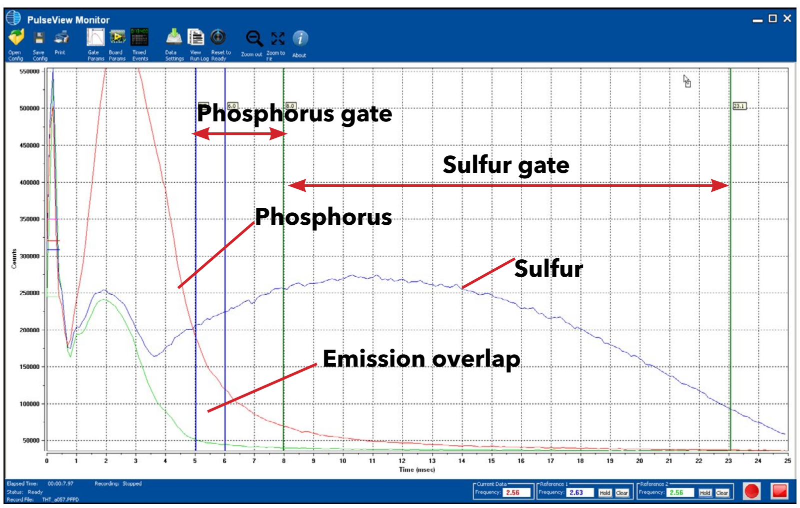


Figure 1.4. Carbon, Phosphorus, and Sulfur Emission Lifetimes

Improve detectivity (see Chapter 13, Defining Detectivity) and selectivity for sulfur and phosphorus by using gated integration that rejects the unwanted CH*, C_2*, and OH* chemiluminescent responses. See Chapter 6, Operation, for further discussion of PFPD operational parameters settings to optimize chemiluminescent responses.

Because the PFPD is a pulsed detector generating pulsed chemiluminescence, its electronics differ significantly from traditional FPD electronics. The PFPD's most basic pulsed electronics consist of a fast electrometer, an event (threshold) detection circuit, and a gated integrator.

A fast electrometer initially amplifies the output from the photomultiplier tube. The subsequent output signal is then digitized and passed through a gated integrator. The operator defines the integration interval by using specified gates. The detector controller then converts this electronic signal to an analog signal that is proportional to the integrated signal.

PulseView Monitor, a Microsoft Windows®-based software package developed by OI Analytical, facilitates the operator's ability to optimize PFPD operation. PulseView Monitor lets the operator set up and optimize all PFPD operating parameters. After these parameters have been uploaded to the detector electronics controller, the software is no longer required for continued PFPD operation. If the chromatographic conditions change dramatically, the operator reactivates PulseView Monitor to program new operational parameters.

Features

- Selectively tuning of the PFPD results in fewer false positive identifications and decreases the need for additional sample preparation.
- Improved sensitivity compared with many standard FPDs enables detecting lower quantities of trace-level contaminants and decreases the need for sample concentration.
- Using only 10% of the gas flow rate of standard FPDs saves operational gas costs.
- Equimolar sulfur chemiluminescence response independent of a compound's molecular structure allows calibration of complex samples.
- Real-time dual analog outputs enable information acquisition for two elements simultaneously.
- Dual gate capabilities optimize selectivity of up to 28 different elements.
- Setting automated timed events for most detector parameters extends the detector's performance range for a given application and often minimizes the need for complicated chromatographic solutions with difficult applications.
- The self-cleaning design prevents soot formation and minimizes maintenance.
- Constant reignition eliminates "flameout" problems caused by water and other solvents, which is common in standard FPDs.
- PulseView Monitor software provides real-time display of the emission resulting from each flame pulse. It facilitates detector setup and configuration, optimizes operating parameters, and troubleshooting without the use of an oscilloscope.
- PulseView Analyzer software provides post-run data processing and analyzer.
-

Specifications

Product Specifications

- Pollution degree: 2
- Installation category: II
- U.S. Patent Number: 5,153,673

Performance Specifications

Detector Linearity

- Sulfur: quadratic in response, linear to approximately 2.4 orders of magnitude (approximately 3 orders of total dynamic range).
- Phosphorus: first order in response over three orders of magnitude.

Detectivity

- Sulfur: <1 pg S/sec
- Phosphorus: <100 fg P/sec

Selectivity at Optimum Detectivity Levels

- Sulfur: >106 S/C (or greater depending on gate settings)
- Phosphorus: >105 P/C
  Adjustable selectivity with a trade-off in detectivity
  Sulfur: <1 pg S/sec
- Phosphorus: <100 fg P/sec

Sensitivity

- Sulfur signal-to-noise: >300 (at 10 pg S/sec elution rate peak-to-peak noise)

Column Flow Range

- Optimum performance: 1–2 mL/minute
- Maximum: 5 mL/minute

Drift

- Sulfur or phosphorus: <10x peak-to-peak noise in 20 minutes

Response Uniformity

- Equimolar ±8% (S, P)

Chromatographic Peak Tailing

- <0.2 sec in S and P

General Specifications

Controller Board Inputs and Outputs

- Two channels (0–1 V)
- One serial RS-485
- One signal in (electrometer; PFPD)
- High voltage out (PMT 0–1,000 V)
- Ignitor current (0–3.1 A)
- S/W HV protection (PMT protection)
- Timed events (from GC start sense): autozero, range, attenuation, ignitor, mode or channel (e.g. S, P, N, C), and record

Temperature Limitations

- Minimum: 180 °C
- Maximum: 420 °C

Carrier Gas

- 5 mL/min maximum flow rate (He, N_2); up to 10 mL/min using H_2 carrier gas

Typical Gas Consumption

- H_2: 10–15 mL/min
- Air: 20–30 mL/min

Heating Mode

- Direct heating by heater cartridge
- RTD (PRT) or thermocouple temperature sensing

Environmental Considerations

- Indoor use only
- Relative humidity; (80% max up to 31 °C, 50% max 32–40 °C)
- Operating Temperature:10–40 °C
- Altitude: Maximum 2,000 m

Controller Dimensions

- Electronics: 6.9" H x 2.4" W x 9.9" D   (17.5 cm H x 6 cm W x 25 cm D)
- Pneumatics: 6.9" H x 2.4" W x 10.3" D   (17.5 cm H x 6 cm W x 27.5 cm D)

Gas Flow Control

- Manual control of detector gases with mass flow controllers and metering valve
- EPC control of detector gases through 00 GC

Options

- Gas Flow Control: automatic electronic control of detector gases
- Combustor: 3 mm
- Filter and PMT Configurations: for 28 different elements

Requirements

Detector Controller Power Requirements

- 115–230 VAC, 50/60 Hz 75VA

Host Software Requirements

- Windows 7, 8, 8.1, or 10 operating system

PC Hardware Requirements for PulseView Software

- Processor: must meet Microsoft® minimum requirements for the operating system.
- SVGA monitor (or higher)
- One USB port (2.0 or higher)

Gas Requirements

- Carrier: helium, nitrogen, or hydrogen, 80 psi, 99.999% purity or better
- Air: 60 psi, zero air, 99.999% purity or better
- Hydrogen: 60 psi, 99.999% or better (electrolytic grade)
- Recommended: hydrocarbon and oxygen filters for carrier gas

Safety Information

The 5383 PFPD Controller meets the European Union Directives for emissions and safety as noted in the Declaration of Conformity for this instrument as tested and documented by a certified independent laboratory. Cleaning is not required for this unit to function properly.

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.

ATTENTION: No user serviceable parts inside. Refer servicing to qualified service personnel.

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 is not liable for the operator's failure to comply with warnings and precautions.

Connect the PFPD 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.**

Any power cord used shall have the same or greater power rating as the cord supplied with the instrument. Position the PFPD controller so there is easy access to the power switch.

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 void the warranty.

- Verify all heated areas have cooled before handling or wear adequate hand protection to prevent burns.

Compressed Gas Cylinder Precautions

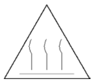
WARNING: Hydrogen is highly flammable and may cause an explosion if it is allowed to build up in an enclosed area, such as in the GC oven. Exercise great care when handling hydrogen. Check all gas fittings periodically for leaks and keep open flames and other sources of ignition clear of the detector.

- 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
- Hydrogen is extremely flammable and is identified as an asphyxiant. Handle and store this gas and the cylinders containing it in a manner consistent with OSHA regulations. Do not bring hydrogen into contact with open flames and easily ignited materials except under approved conditions controlled by the analyst. Maintain adequate ventilation in areas where this material 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 are 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 is 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.

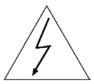
Safety Symbols

The following symbols may be located on the instrument:

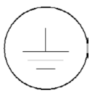
Warning/Caution, see accompanying instruction for more information.



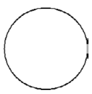
Indicates a hot surface.



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

5383 Detector Assembly, Assembled View

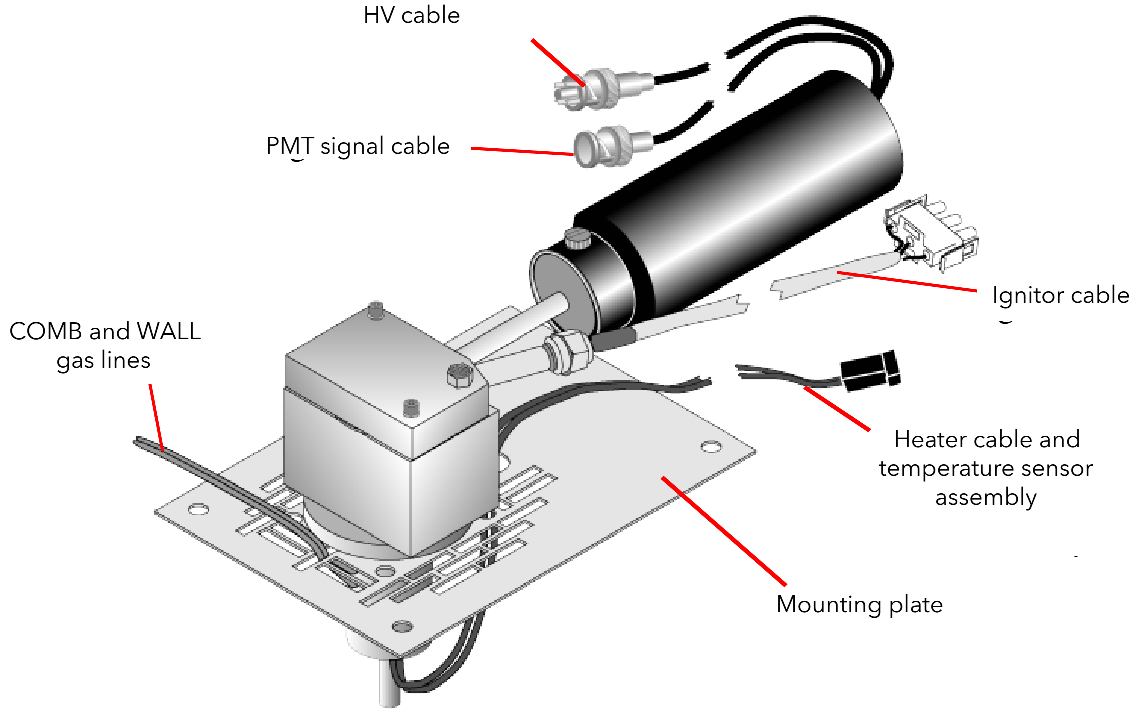


Figure 2.1. 5383 Detector Assembly, Assembled View

HV cable (SHV connector) connects to the PMT HV (high voltage) connector on the back of the 5383 detector controller.

Ignitor cable connects to the Ignitor connector on the back of the 5383 detector electronics controller.

PMT signal cable (BNC connector) connects to the PMT signal connector on the back of the 5383 detector electronics controller.

5383 Detector Assembly, Exploded View

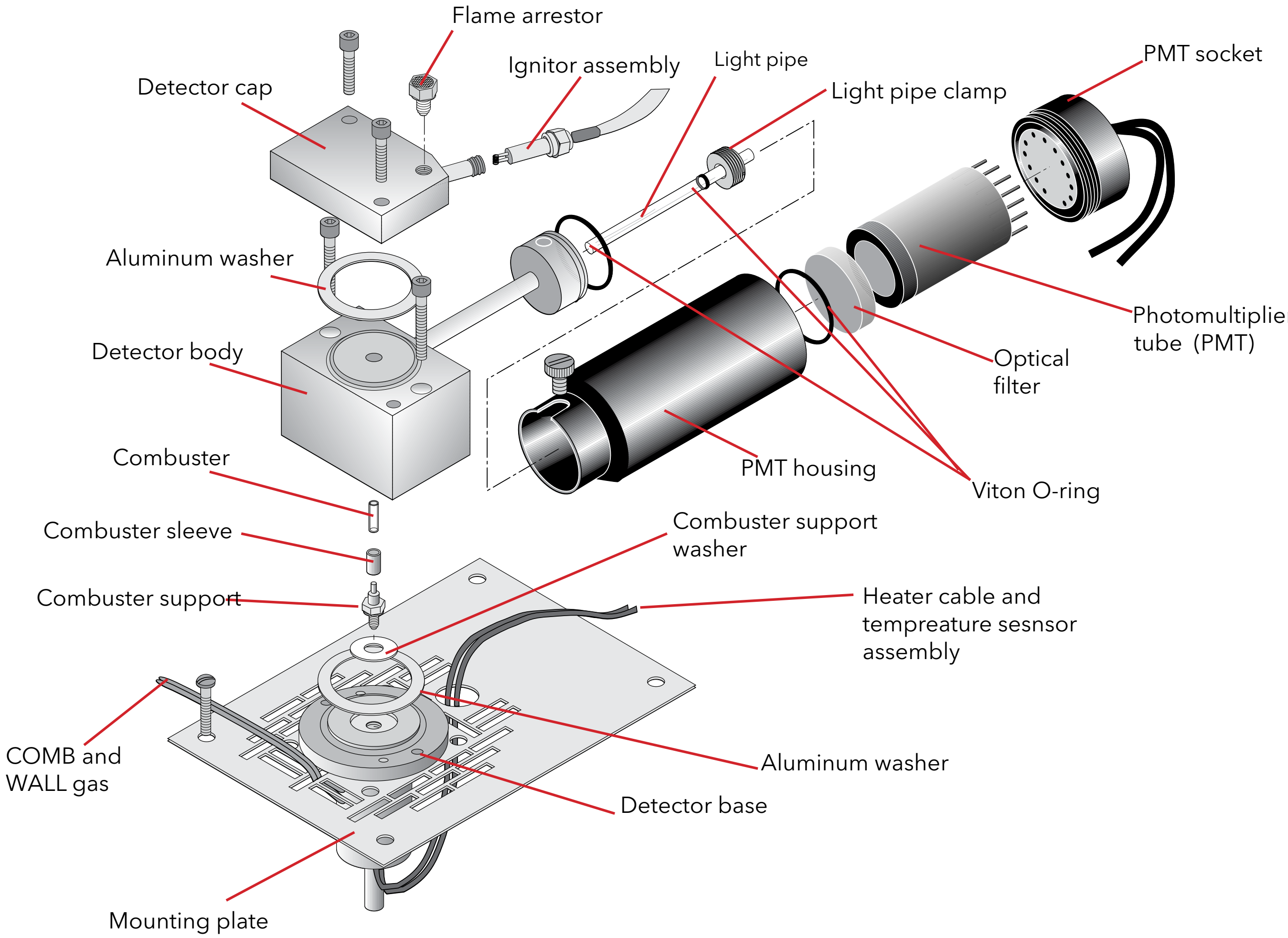


Figure 2.2. 5383 Detector Assembly, Exploded View

Aluminum washers create a gas-tight seal between the detector cap and the detector body and between the detector body and detector base.

Combustor is a transparent quartz tube within which the GC column effluent combusts.

Combustor support mounts into the base with the combustor support washer. The combustor support positions the combustor at the proper position for optimum response.

Combustor support washer creates a gas-tight seal between the combustor support and the base, and isolates the wall gas from the combustor gases.

Detector base contains the combustor support and the heater and temperature sensor.

Detector body contains the combustor and supports the light pipe and the PMT housing assembly.

Detector cap provides an attachment for the ignitor assembly and provides the path to the exhaust vent.

Flame arrestor prevents the flame from propagating out through the vent.

Gas lines connect to the detector base. The lower gas line (COMB) provides the hydrogen-rich combustor gas that sweeps out the combustor base. The upper gas line (WALL) provides an oxygen-rich gas mixture that sweeps out the region between the detector body and the combustor, providing gas to the ignitor.

Heater cable and temperature sensor assembly lets the host GC control the temperature of the PFPD base.

Ignitor assembly threads onto the detector cap's ignitor tube.

Light pipe is a quartz rod specifically designed for optimal chemiluminescence transmission to the PMT.

Light pipe clamp compresses the light pipe O-ring, which provides a gas-tight seal between the light pipe and the detector body.

Mounting plate attaches the PFPD assembly to the top of the GC oven using mounting screws. (The mounting plate is GC-specific.)

Optical filter mounts inside the PMT housing. An O-ring keeps the filter from moving and ensures that all the light from the light pipe passes through the optical filter. The filter spectrally isolates specific chemiluminescence.

Photomultiplier tube (PMT) converts light energy into electrical current. Increasing the voltage applied to the PMT increases the current generated by the PMT.

PMT housing contains the optical filters, PMT, PMT socket, and the electronic cables for the signal and high voltage of the PMT.

PMT socket attaches to the PMT connector pins. Voltage supply to the PMT and electrical responses to chemiluminescence (resulting from flame propagation through the combustor) from the PMT are transmitted through these pins.

Viton® O-ring provides a light-tight seal between the PFPD detector body and the PMT housing-filter assembly. It prevents optical filter movement, light leakage around the optical filter, and provides a soft bed for the PMT. The Viton O-ring around the light pipe holds the light pipe in place when the light pipe clamp is tightened.

Model 5383 Detector Pneumatics Controller, Front View

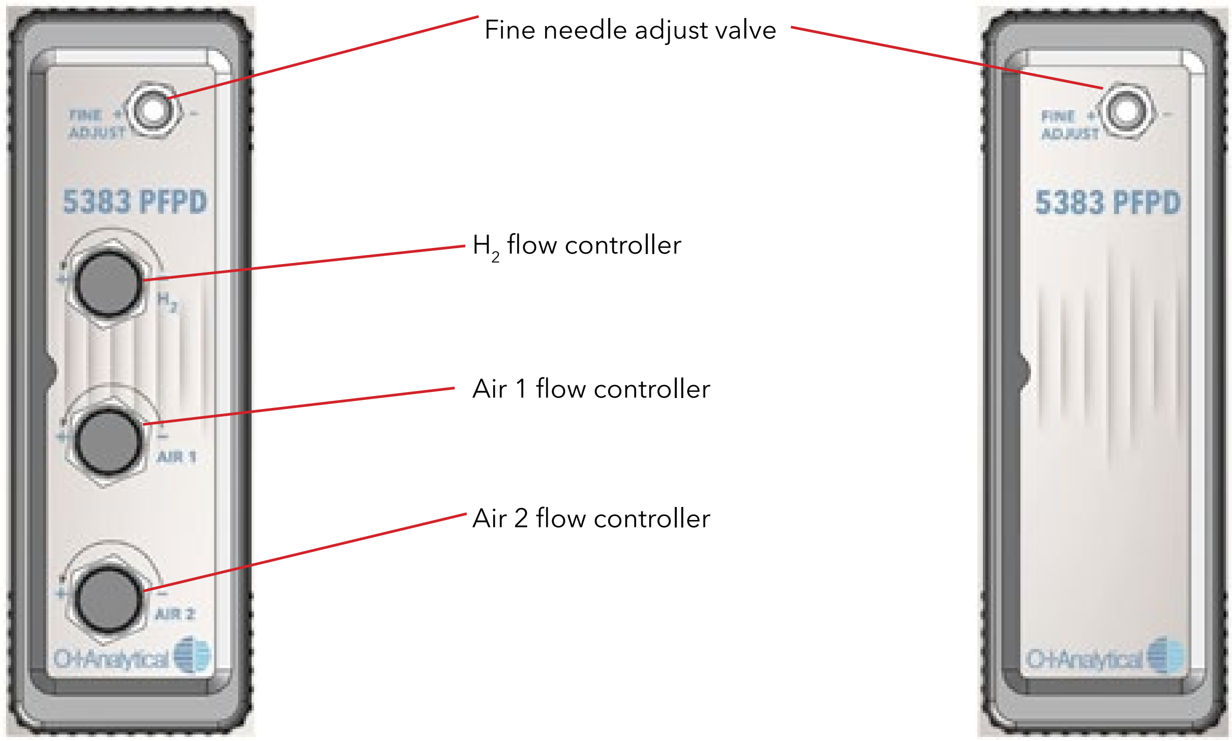


Figure 2.3a. Model 5383 Detector Pneumatics Controller with Manual Gas Flow Control

Figure 2.3b. Model 5383 Detector Pneumatics Controller, EPC Ready for GCs with Electronic Gas Flow Control

Pneumatic Controls

Air 1 flow controller controls the air flow to the combustor.

Air 2 flow controller controls the air flow to the external wall of the combustor and ignitor volume.

Fine adjust needle valve balances the flow of Air 1 and H_2 between the combustor and the wall flow paths.

H_2 flow controller controls the amount of hydrogen added to the COMB and WALL Gases.

Model 5383 Detector Pneumatics Controller, Back View

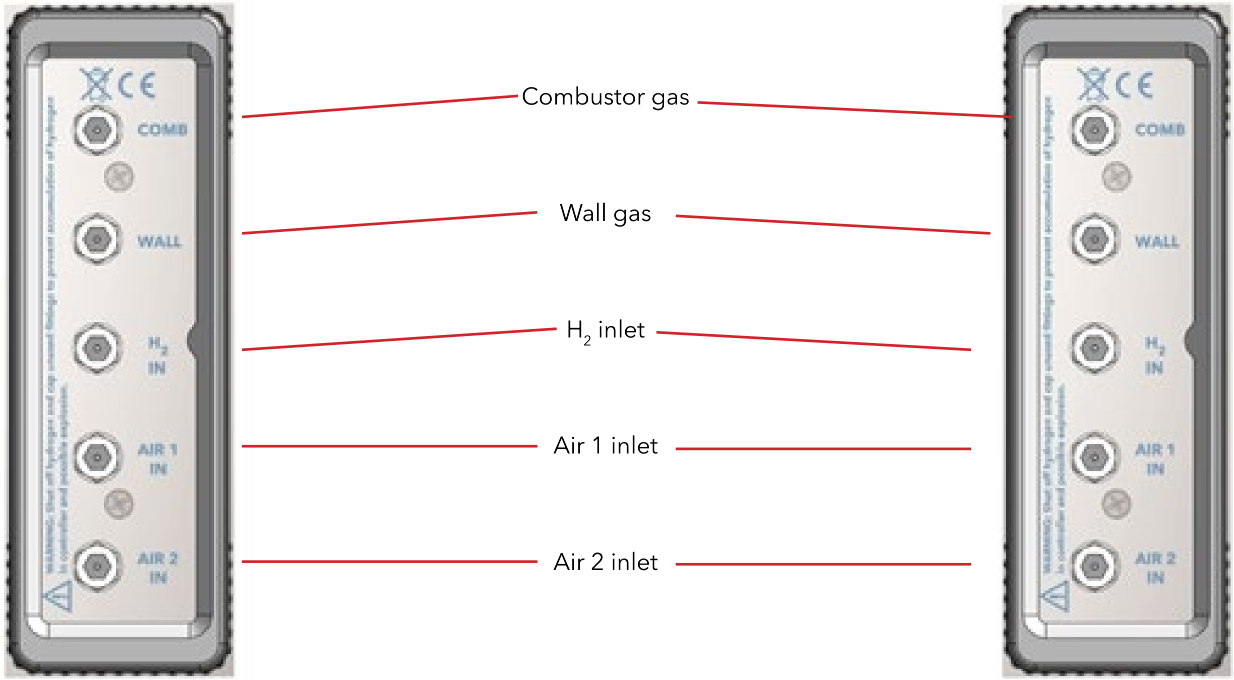


Figure 2.4a. Model 5383 Detector Pneumatics Controller with Manual Gas Flow Control

Figure 2.4b. Model 5383 Detector Pneumatics Controller PC Ready for GCs with Electronic Gas Flow Control

NOTE: All gas inlets have a 60 PSI (400 kPa) maximum.

Gas Connections

Combustor gas connector attaches to the combustor gas line (COMB) from the detector pneumatics controller to the detector base.

Detector pneumatics controller to the detector base.

Wall gas connector attaches to the wall sweep gas line (WALL) from the detector base.

H_2 inlet connector attaches the hydrogen supply to the detector pneumatics controller.

Air 1 inlet connector attaches the air supply to the detector pneumatics controller for manual pneumatics. The connector attaches to the Air 1 supply for EPC-ready pneumatics.

Air 2 inlet connector attaches the Air 2 supply to the detector pneumatics controller, only for EPC-ready pneumatics.

NOTE: The Air 2 supply is plugged for the manual gas flow control model.

Model 5383 Detector Electronics Controller

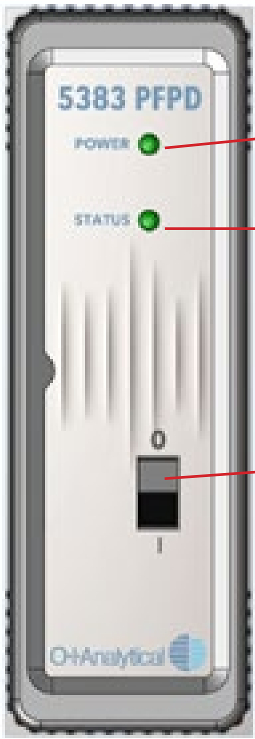


Figure 2.5a. Model 5383 Detector Electronics Controller, Front View

Figure 2.5b. Model 5383 Detector Electronics Controller, Back View

Electrical Indicators and Switches (Front Panel)

Power LED (green) indicates the power status of the electronics unit.

Status LED (red) indicates an error exists (if on).

Power switch is used to enable power to the electronics module (0=off; 1=on).

Electrical Connections (Back Panel)

PMT HV connector (SHV connector) attaches the PMT HV (high voltage) cable to the detector electronics controller.

Ignitor connector (three-pin connector) attaches the ignitor assembly cable from the PFPD to the detector electronics controller.

Channel 1 connector (BNC connector) attaches the cable that transmits the channel 1 output signal from the detector electronics controller to the data handling device.

Channel 2 connector (BNC connector) attaches the cable that transmits the channel 2 output signal from the detector electronics controller to the data handling device.

PMT signal connector (BNC connector) attaches the PMT Signal cable from the PFPD detector to the detector electronics controller.

RS-485 port (RJ-11 connector) attaches the detector electronics controller to the PC's USB port to operate PulseView Monitor software.

Power receptacle is an International Electrotechnics Convention (IEC) type power inlet receptacle.

Remote start connector attaches a remote start cable from the GC to the detector controller to synchronize the PFPD timed events with the start of the GC's analytical run.

Chapter 3 Installation

Chapter 2 defined the names and functions of the various components of the 5383 PFPD. Refer to the names in that chapter for components used to install the 5383 PFPD onto the gas chromatograph (GC).

NOTE: The PFPD must be installed by a qualified technician, preferably an OI Analytical-certified 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 detector operation is verified.

NOTE: All instruments that are returned to OI Analytical for service or warranty repair must be shipped in the instrument's original box with its packing material. For proper shipping materials, contact the Order Entry Department at (800) 336-1911 or (979) 690-1711.

Remove the following seven components:

- detector assembly containing an attached PMT housing assembly, two gas lines, heater assembly cable, ignitor assembly cable, and detector mounting plate;
- detector electronics controller containing the PFPD controller PCB board;
- detector pneumatics controller containing the gas mixing assemblies;
- PFPD startup kit;
- PFPD GC/Pneumatics startup kit;
- detector electronics controller startup kit; and
- CD containing the PulseView software and user manuals.

Proper storage and handling of the detector components ensures reliable assembly and disassembly of the PFPD. Never handle combustor tools or forceps parts that touch the internal parts of the PFPD without latex gloves. Use clean laboratory tissues, the Teflon® combustor extractor, or Teflon-coated forceps to remove or replace internal detector parts. Place removed parts on a clean, lint-free laboratory tissue or in a clean glass beaker.

Installing the 5383 PFPD Detector Assembly in the Agilent 6890/7890 GC

Mount the PFPD assembly on an Agilent 7890, Agilent 6890 GC, or other GC model. The following description focuses on the PFPD installation procedure for the **Agilent 6890 and 7890 GCs**. If installing the PFPD in the **Agilent 6890 Valve Box**, see Installing the 5383 PFPD in the Agilent 6890/7890 Valve Box, in this chapter. If installing the PFPD onto a **Shimadzu GC-2014**, see Installing the 5383 PFPD Detector Assembly onto the Shimadzu GC-2014 in this chapter.

To mount the detector on other GC models, refer to the appropriate GC user manual, or specific instructions included with the PFPD startup kit. Any other service requests, contact OI Analytical Technical Support at (800) 336–1911 or (979) 690–1711.

Preparing the GC

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

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

2. If the GC was previously installed, turn off the GC power supply and unplug the power cord. Turn off the supply gases at their sources after the heated zones have cooled.

WARNING: Detectors may be hot!

3. Remove the GC detector cover, the electronics compartment top and right slide covers, the top back cover and metal RFI shield beneath it, and the back metal cover.

4. If a detector is already in the desired detector port for the PFPD, allow the installed detector to cool. Turn off and disconnect all associated gas lines, GC columns, and electronics, and remove the detector according to the proper procedure described in the detector operator's manual.

5. If no detector was previously installed in the preferred detector port, remove the plastic cutout to the right of the selected port (Figure 3.1).

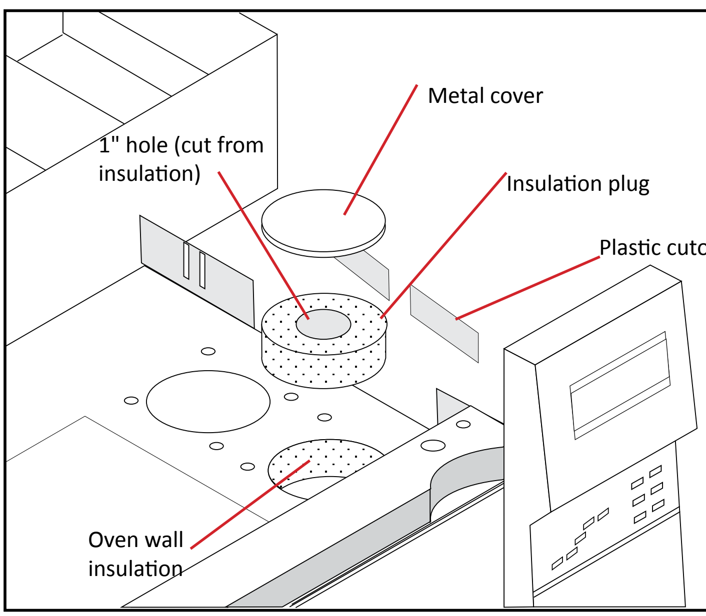


Figure 3.1. Preparing the GC detector port

6. Remove the circular metal detector port cover and the circular insulation plug from the desired mounting position in the GC's detector area. Set the insulation plug aside for later use.

7. Cut a hole in the top-right GC cover (Figure 3.2).

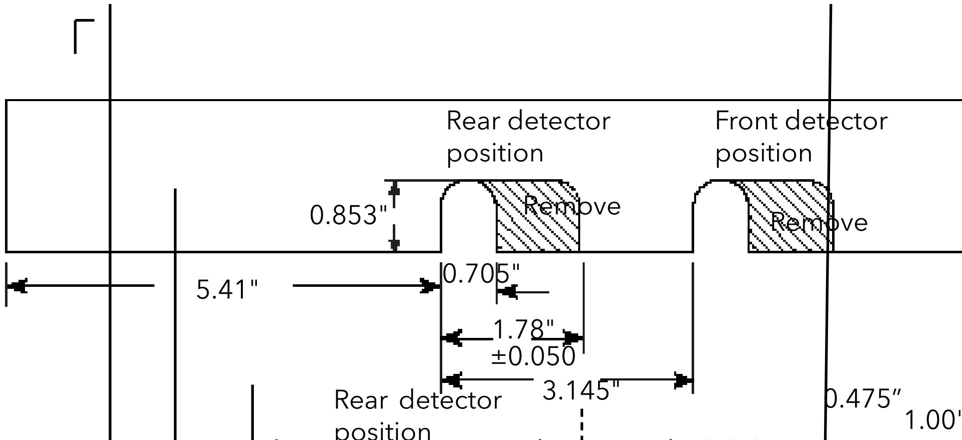


Figure 3.2. Preparing the Top-Right GC Cover

8. Cut a hole in the top GC cover (Figure 3.3).

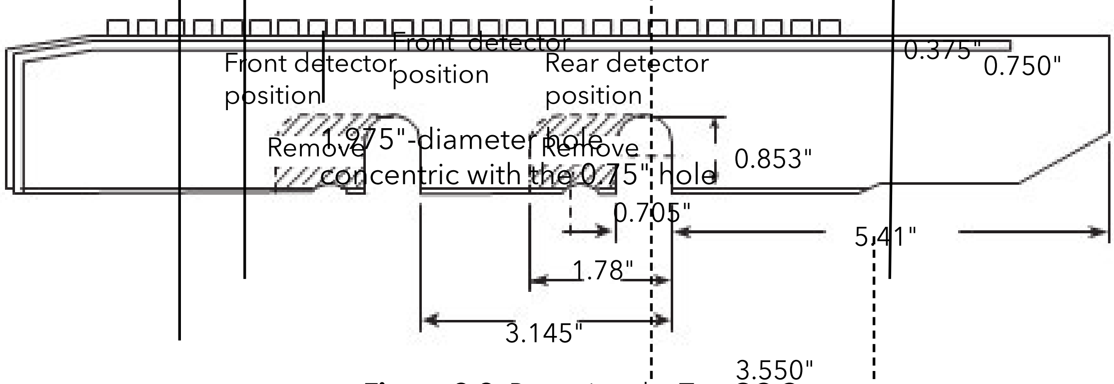


Figure 3.3. Preparing the Top GC Cover

9. Optional: Cut a 1.975"-diameter hole in the top of the GC cover concentric with the existing hole (refer to Figure 3.4).

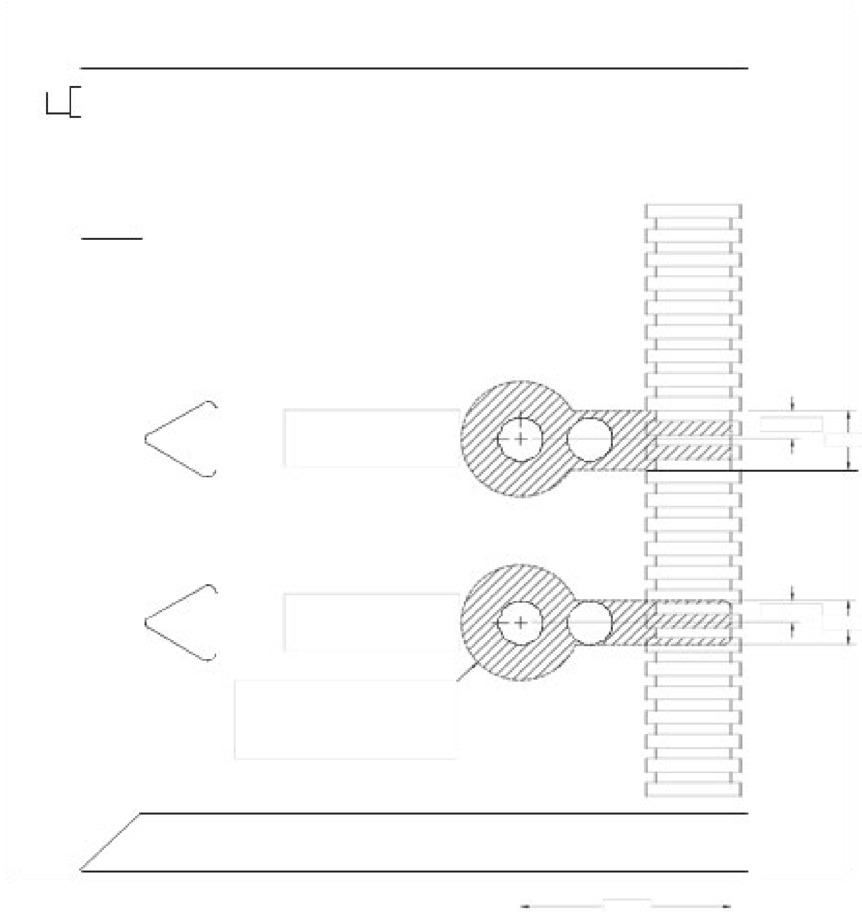


Figure 3.4. Agilent 6890 or 7890 GC Cover Modifications for Mounting the PFPD

NOTE: A possible build-up of heat may cause discoloration and/or minor deformation to the inside of the GC cover. While this step prevents the cosmetic damage by providing a location for heat to dissipate, the unit will still operate safely without the extra hole.

10. Place a paper towel or tissue paper on the floor of the GC oven. From inside the oven, cut away the oven insulation covering the detector port. The oven insulation covering to be cut away is visible though the opening in the metal oven lining that corresponds to the detector port. After cutting away the insulation, carefully remove all the insulation material debris and the paper towel.

11. Cut a hole approximately 1" (25 mm) in diameter in the center of the previously removed insulation plug so that the center of the hole lines up with the center of the detector port. Replace the insulation plug in its original position in the detector port.

12. Install the appropriate analog input boards (if required) into the GC. (See the *Agilent 6890 or Agilent 7890 Series Gas Chromatograph Operator's Manual.*)

Mounting the PFPD on the GC

1. Carefully remove the PFPD assembly from its packaging.

2. Before installing the PFPD onto the GC, ensure the following components are attached to the detector assembly (refer to Figures 2.1 and 2.2).

    - Check that the ignitor assembly (PN 282624) is attached to the detector cap. If not, remove the protective cap from the ignitor connector on the detector cap. Remove the ignitor assembly from its protective cover. Do not touch the ignitor coil. Carefully insert the ignitor assembly into the ignitor housing and tighten the hexagonal nut (located behind the ignitor) onto the ignitor connector.

    - Check that the heater cable assembly is embedded in the detector base. If not, thread the heater cable assembly down through the round opening in the detector mounting plate.

    - Insert the heater element (larger diameter, metal probe) and temperature sensor (smaller diameter, white porcelain probe) into the appropriate receptacles in the bottom of the detector base. Do not bend the wire leads protruding from the heater element and temperature sensor at the base of the detector.

    CAUTION: Excessive bending of the wire leads on the heater or temperature sensor will cause damage.

    - Check that the column inlet fitting with no-holed ferrule is installed at the bottom of the detector base. If not, insert a 1-16" no-holed ferrule (PN 197079) into the GC column nut (PN 223057) with the tapered end of the ferrule facing into the nut. Gently screw the column nut into the bottom of the detector base to prevent debris from penetrating the detector base during installation.

3. Evenly pack a 1/4" thick strip of the insulation blanket (PN 280610) around the detector base below the mounting plate so none of the detector base remains exposed. Do not wrap the heater cable with the insulation.

4. Carefully press the PFPD assembly and surrounding insulation into the previously hollowed-out insulation plug in the selected detector port (Figure 3.5 for Agilent 6890 and Figure 3.6 for Agilent 7890). Do not excessively bend the leads protruding from the heater element and temperature sensor. Ensure the detector base protrudes into the GC oven cavity and that the detector mounting plate is flush with the metal cover in the detector area.

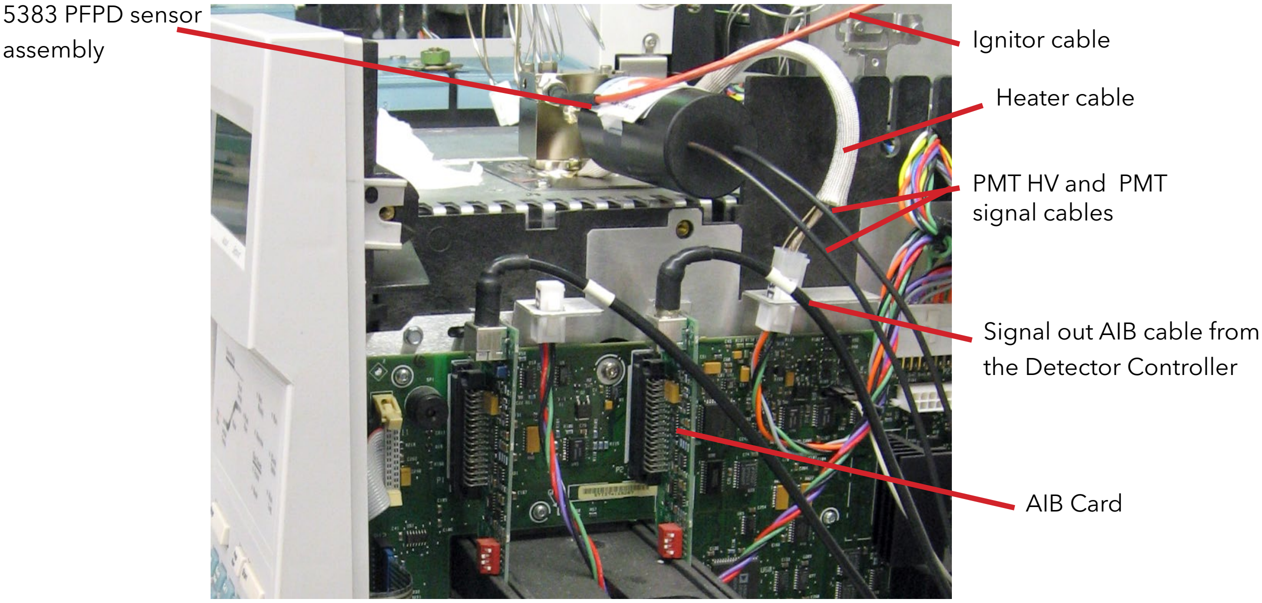


Figure 3.5. Placing the PFPD in the Agilent 6890 and attaching the heater cable

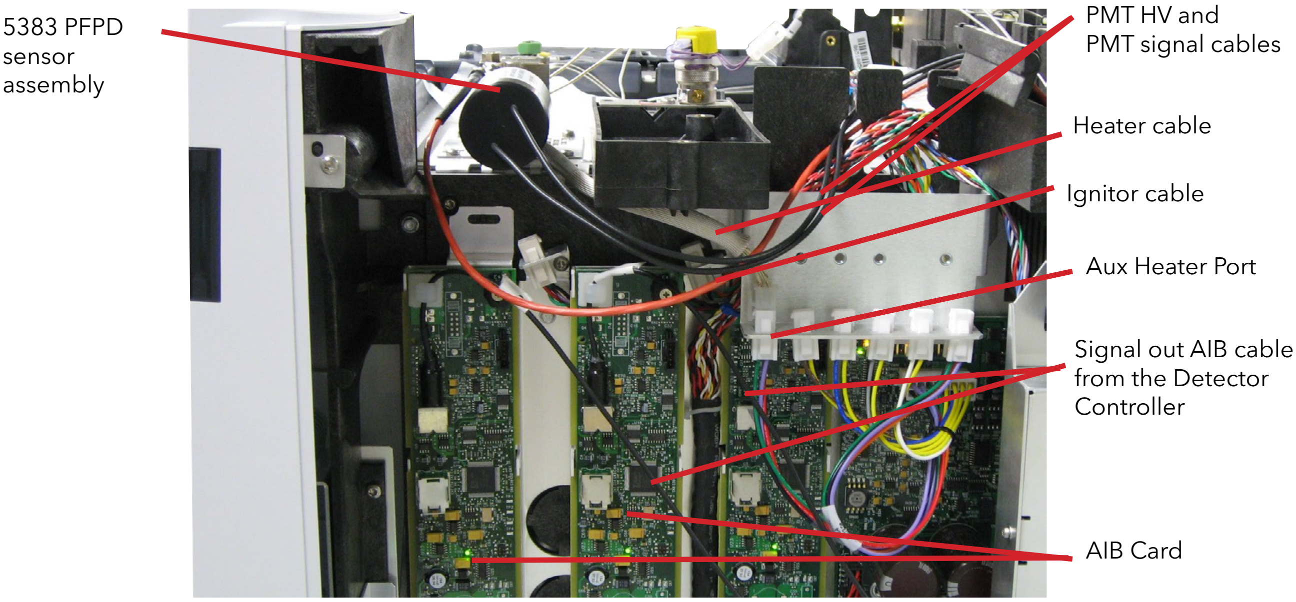


Figure 3.6. Placing the PFPD in the Agilent 7890 and Attaching the Heater Cable

5. Align the detector mounting plate with the screw holes corresponding to the selected detector port. Secure the mounting plate using the mounting screws (PN 252585 for the Agilent 6890 and 7890).

NOTE: When mounted, the PMT protrudes to the right side of the GC. If this detector orientation needs changing, refer to Installing the Combustor and Reassembling the PFPD in this chapter.

Installing the Aux Temperature Zone Bracket in the Agilent 7890

Only required if using 7890 Aux EPC module. Not required if using 7890 OIM module.

1. Carefully cut the nylon Ty-Rap® holding the PFPD cables and gas lines together.

2. Do not remove the nylon tags designating the combustor and wall gas lines. Unroll the gas lines without kinking them.

3. Screw the Aux Temperature Zone Bracket (valve driver bracket) into the right side of the GC using the two captive screws. Refer to Figure 3.6 for the location.

4. Plug the connectors on the valve driver cable harness up through the slots on the valve driver bracket.

5. Plug the larger 2 x 2 heater sensor connectors (P1,P2) into the outside slots and the smaller 1 x 2 valve driver connectors (P3 to P6) into the four middle slots (refer to Figure 3.7).

6. Plug the other end of the cable into the P22 connector on the GC Main Board.

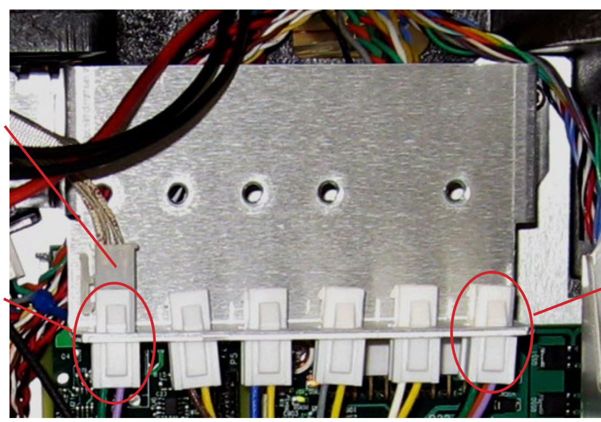


Figure 3.7. Installed Aux Temperature Zone Bracket

Installing the 5383 PFPD in the Agilent 6890/7890 Valve Box

Install the 5383 PFPD in the Agilent 6890 GC valve box area using the following instructions. The valve box sits on top of the Agilent 6890 and contains the valves and plumbing, heated zones, sensors, and insulation. For more information, see the *Agilent 6890 Series Gas Chromatograph Operator's Manual*.

1. Cut out and remove the valve box plate (Figure 3.8).

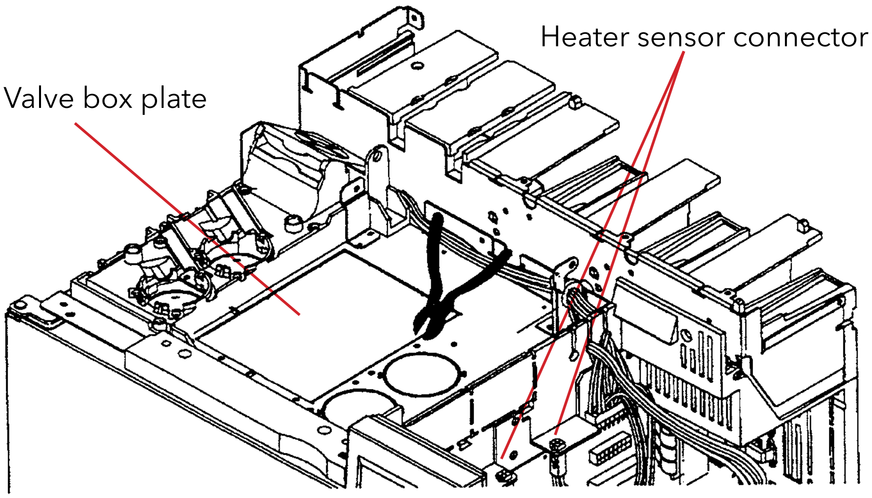


Figure 3.8. Valve Box Plate

2. Remove the precut insulation layer.

3. Place a paper towel or tissue paper on the floor of the GC oven. From inside the oven, cut away the insulation covering the front valve port (Figure 3.9). The front valve port aligns with the hole on the PFPD valve box mounting plate (PN 296350). After cutting away the insulation, carefully remove all the insulation material debris and the paper towel.

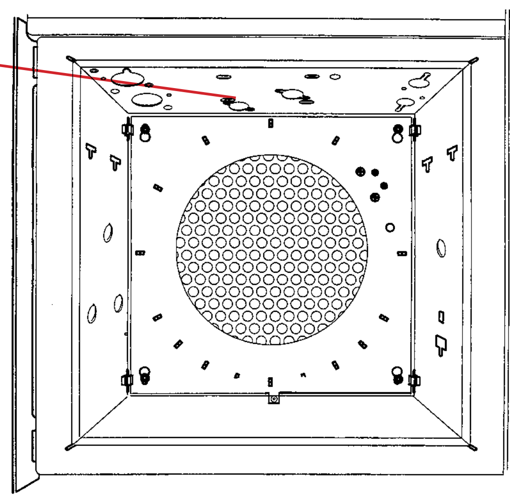


Figure 4.9. Inside the Agilent 6890 GC Oven

4. Cut a hole approximately 1" (25 mm) in diameter in the previously removed insulation layer so that the center of the hole lines up with the center of the previously cut detector port hole. Replace the insulation cutout.

5. Place the PFPD valve box mounting plate on the GC. Line up the holes in the plate with the two PEM® nuts located on the GC.

6. Using two Torx® screws (M4 x 10) (PN 252585), attach the valve box mounting plate to the GC.

7. Align the PFPD mounting plate with the four threaded studs on the valve plate. Connect the PFPD mounting plate using four #6 washers (PN 132589), lock washers (PN 132654), and nuts (PN 132).

8. Carefully cut the nylon Ty-Rap holding the PFPD cables and gas lines together. Do not remove the nylon tags designating the combustor and wall gas lines. Unroll the gas lines without kinking them.

9. Connect the cable heater-sensor extension (PN 296368) to the existing heater cable. Connect the other end of the heater cable to the corresponding four-pin nylon sensor connector located in the electronics area above the GC motherboard (Figure 3.8).

10. If both detector ports are occupied by existing detectors, plug the heater cable assembly to the valve heater driver for the GC. If the valve heater driver is installed on the GC, plug the heater cable assembly to either the P1 or P2 position. (Figure 3.10). If the valve cable assembly and bracket are not already installed on the GC, order them separately. Both the valve driver block (PN 280818; Agilent PN G1580-00070) and the valve driver wire harness (PN 280834; Agilent PN G1530-60660) are needed. See the *Agilent 6890 GC Service Manual* for instructions on installing these items.

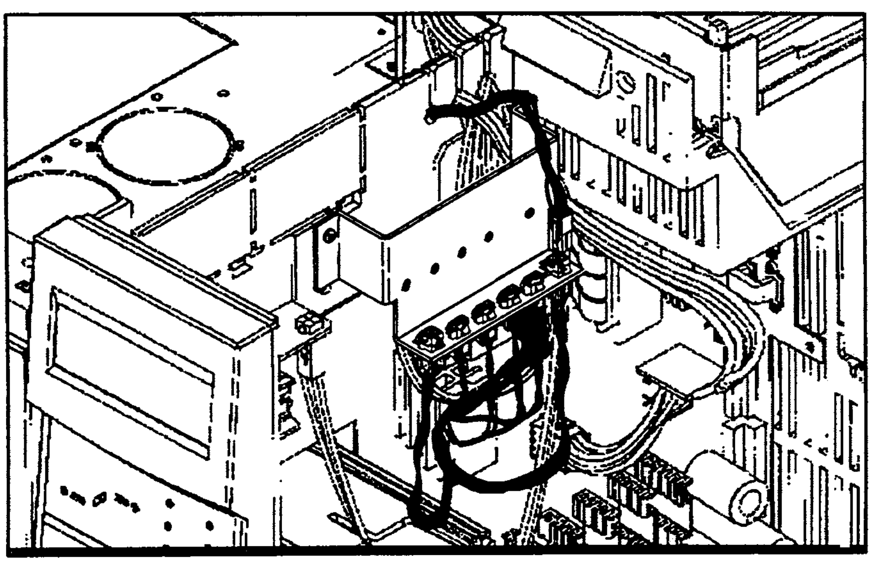


Figure 3.10. Valve Heater Driver

11. Pack the insulation around the detector base to minimize dead space and heat loss.

Installing the 5383 PFPD Detector Assembly onto the Shimadzu GC-2014

Install the Model 5383 PFPD onto a Shimadzu GC-2014 using the following instructions. For more information, see the *Shimadzu GC-2014 Operator's Manual*.

Preparing the GC

1. Turn off the GC's power supply and unplug the power cord. Turn off the supply gases at their sources after the heated zones have cooled.

2. Remove the GC's top cover. (Refer to the *Shimadzu GC-2014 Operator's Manual*.)

3. The back-right detector port on the Shimadzu GC's top plate is the preferred port for the PFPD (see Figure 3.11). This location ensures that the photomultiplier tube (PMT) will not receive excess heat from the injector port.

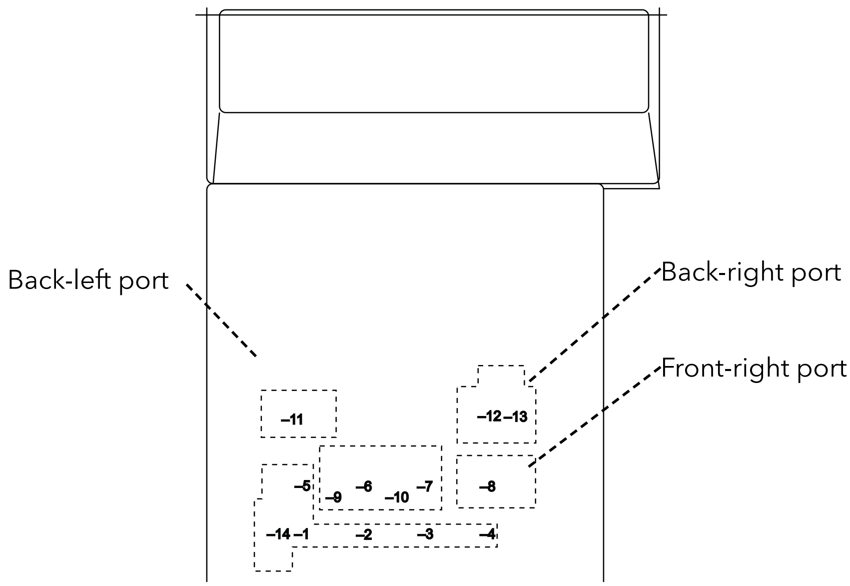


Figure 3.11. Shimadzu GC-2014 Detector Mounting Ports

NOTE: The PFPD can be installed into the front-right or back-left port, as long as the PMT housing does not come too close to an injector or another heated detector. Avoid running the heater cable alongside the PMT signal cable. Either condition will contribute to noise to the detector signal (see Figure 3.11).

4. After selecting the desired detector port, cut a 1" diameter x 0.5" deep circle out of the GC insulation of the desired detector port (see Figure 3.12).

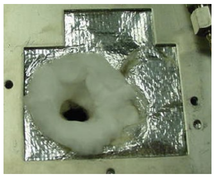


Figure 3.12. Cut into the Insulation

Mounting the PFPD on the GC

1. Verify the heater and Platinum Resistance Temperature (PRT) device are installed into the PFPD base.

2. Orient the PFPD so the PMT housing is facing the left side of the GC (back-right or front-right ports).

3. To ensure proper heating, the PFPD base should fit snugly into the GC insulation. If not, wrap some insulation (PN 280610, provided in the startup kit) around the base of the PFPD.

4. Place the PFPD into the detector port.

5. Attach the cable clamp (PN 321904, provided in startup kit) to the top of the GC bracket (as shown in Figure 3.13).

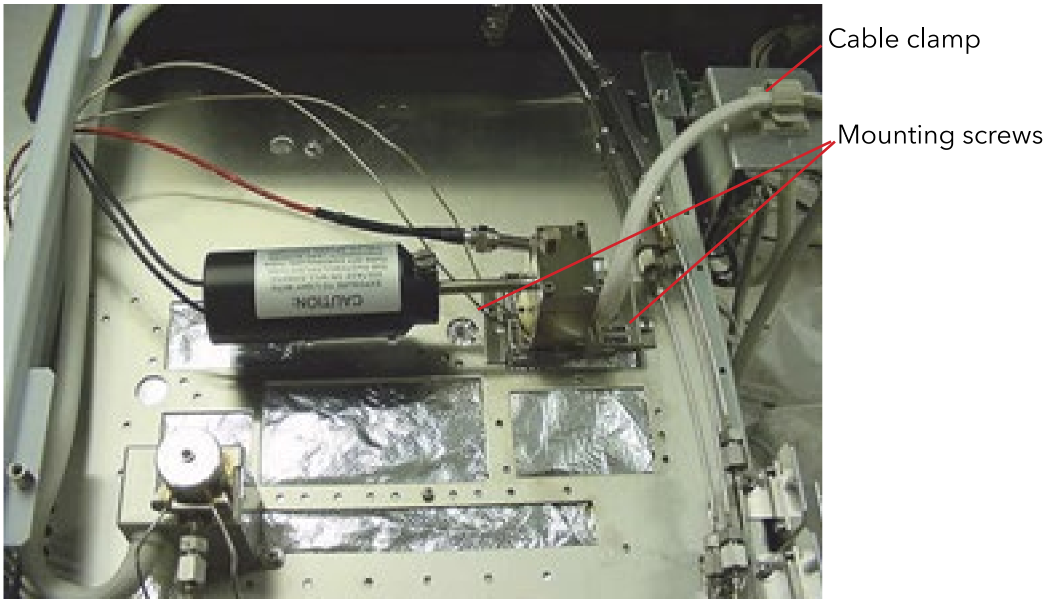


Figure 3.13. Mounting Screw Position

6. Route the heater cable to the right of the instrument through the cable clamp.

7. Place the PMT and ignitor cables to the left side of the GC.

8. Route the tubing:

   - For manual control of the gas flow lines, route the tubing to the left side of the GC.
   - If operating the PFPD with the Shimadzu Aux APC module, route the tubing toward the back of the GC.

9. Attach the PFPD mounting plate to the GC top plate using the two screws provided in the startup kit (PN 169963). Use Figure 3.13 as a reference for the position of the mounting screws.

10. Run the PMT cable, the ignitor cable, and the wall and combustion gas lines through the access hole in the left side of the GC (refer to Figure 3.14).

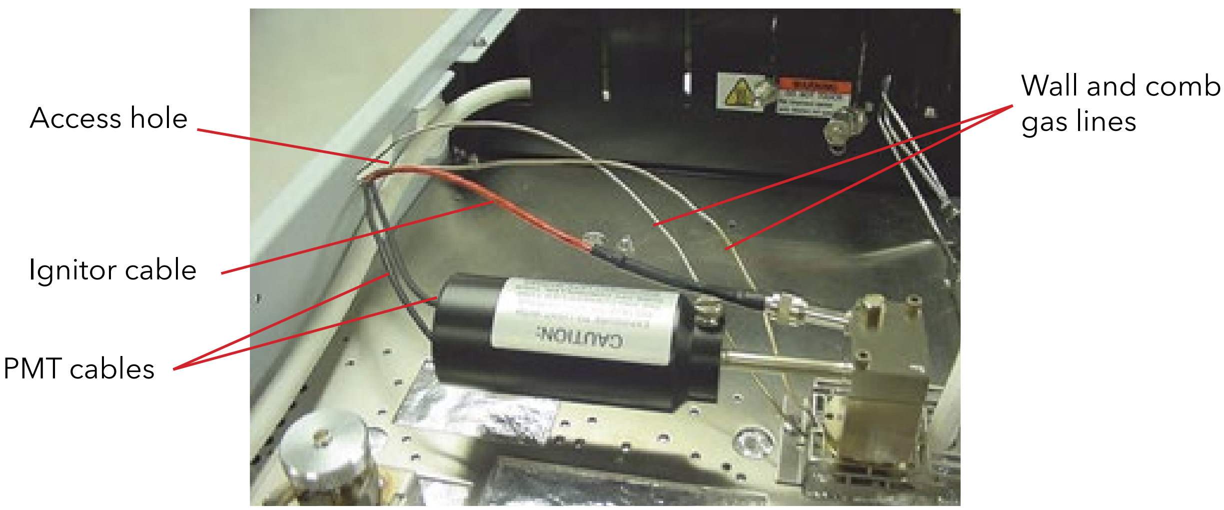


Figure 4.14. Routing the Cables and Combustion Gas Lines

Installing the 5383 Detector Controller

Setting up the Detector Controller

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

All GC models

1. Set the detector controller on the laboratory bench next to the GC.

   Because the detector ports in most GCs are located on the right side, place the detector controller to the right of the GC; however, place the detector controller on the left side of the Shimadzu GC-2014.

2. Ensure the detector controller power switch on the back panel is turned off.

3. Plug the appropriate end of the power cord (PN 116038) into the power receptacle on the back of the detector controller.

4. Depending on the detector controller specifications, plug the other end of the power cord into an appropriate power outlet.

Installing the Heater 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 megohm resistor to an appropriate earth ground.

Agilent 6890 or 7890

1. 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 3.5) or Agilent 7890 (Figure 3.6).

2. For a 7890 equipped with an Aux EPC module, plug the PFPD heater cable into the Aux temperature heater port (Figures 3.6 and 3.7).

Agilent 5890

1. Disconnect the brown plug J10 on the GC motherboard (GC right side) and remove the clear plastic housing.

2. Unplug the brown plug J9 on the far right of the motherboard by squeezing its two locking levers. Press the two heater pins (large diameter) into the GC receptacles corresponding to the selected detector port (DET A and DET B correspond to the front and back detector ports, respectively). Push plug J9 back into its original position on the motherboard.

3. Unplug the black plug P7 in the upper-right corner of the motherboard, and press the two temperature sensor pins (small diameter) into the GC receptacles corresponding to the selected detector port (DET A and DET B correspond to the front and back detector ports, respectively). Push plug P7 back into its original position on the motherboard.

Shimadzu GC-2014

1. Route the heater/PRT cable through the cable clamp and to the detector heater board.

2. Plug the heater cable into the connection labeled DET2 for the back detector position (see Figure 3.15)

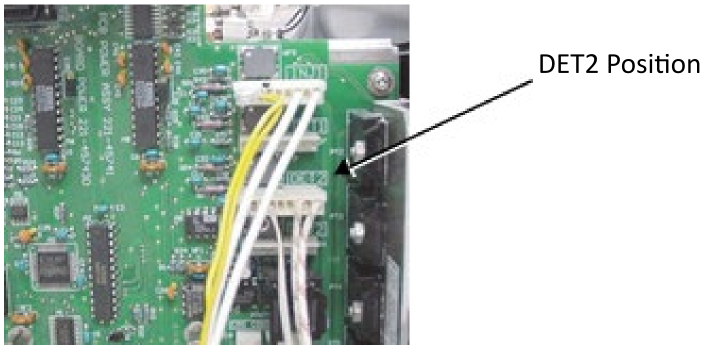


Figure 3.15. Routing the Heater Cable

All Other GC Models

Consult the appropriate GC User Manual for instructions on making the heater cable and sensor connections.

Installing the Ignitor and PMT Cable Connections

All GC Models

NOTE: The PMT housing must be as far away from other heated zones (injector, other detectors, etc.) as possible. Route the heater cable away from the PMT and signal cables. Routing alongside the signal or heater cables may contribute to detector noise. Preferred PFPD orientations:

- Route the ignitor and PMT cables to the back or side of the GC and out to the detector controller.
- Connect the PMT HV cable leading from the PMT housing assembly to the PMT HV connector on the back of the detector controller (see Figure 2.4).
- Connect the PMT signal cable leading from the PMT housing assembly to the PMT signal connector on the back of the detector controller (see Figure 2.4).
- Connect the ignitor assembly cable (PN 282624) leading from the detector cap to the three-pin ignitor connector on the back of the detector controller (see Figure 2.4).

Installing the Signal Cable Connections

Agilent GC Models

1. Connect the appropriate signal out cable to the channel 1 and channel 2 connectors on the back of the detector controller.
2. Connect the opposite end of this cable to the appropriate connector on the chromatographic data system signal input (see Figures 3.5 and 3.6 for the AIB connector).

NOTE: See Chapter 8 Replacement Parts for the part numbers of the signal output cables.

Shimadzu GC-2014

1. Run the signal cable(s) for PFPD channel 1 and channel 2 from the back of the 5383 controller to the appropriate data handling device.

2. If using the Shimadzu 2014 A/D card(s), install the card(s) per the Shimadzu instructions.

3. Attach the terminal strip (provided with the A/D card(s)) to the back of the GC.

4. Connect the spade lug end of the signal cable(s) to the terminal strip, the red wire to"+", and the black wire to "–". See Figure 3.16.

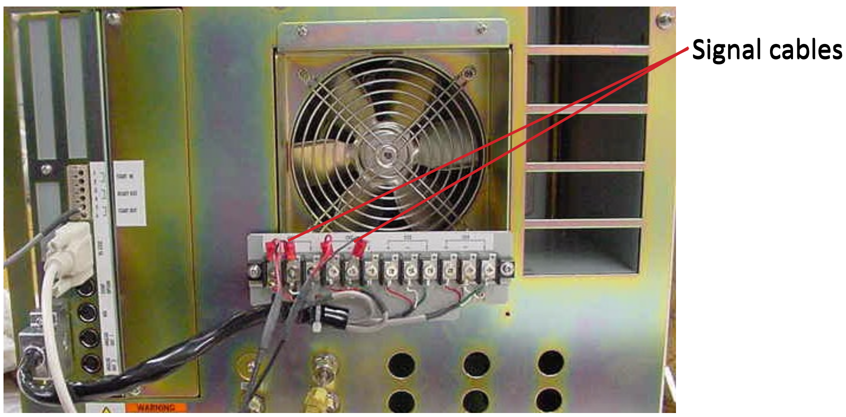


Figure 3.16. Connection Using Two Signal Cables

NOTE:
See Chapter 8, Replacement Parts for the part numbers of the signal output cables.

All other GC Models

Consult the appropriate GC User Manual for instructions on making the signal cable connections.

Installing the Remote Start Cable Connections

All GC Models

1. Connect the three-pin male connector on the remote start cable (PN 288902 or PN 288977, included in the PFPD startup kit) to the remote start connector in the back of the detector controller (Figure 2.4).

2. Connect the other end of this cable to the GC's remote start connector.

Agilent 6890 or 7890

1. For the Agilent 6890 or 7890, connect the two-pin female connector on the remote start cable to the two-pin male "VLV 8" connector on the external event cable (PN 252569, included in the PFPD startup kit) that plugs into the eight-pin MINI-DIN connector marked "EXT EVNT," located on the back of the GC.

2. Program valve 8 as an "External Start" event in the Agilent 6890 or 7890 GC (see the *Agilent 6890 or Agilent 7890 Series Gas Chromatograph Operator's Manual*).

Shimadzu GC-2014

1. Plug the 5383 remote start cable into the 5383 controller.

2. Connect the other end of the remote start cable as follows:
   - Red wire to pin 1 (top pin labeled 5 in Figure 3.17)
   - Black wire to pin 2 (second pin in group 5 in Figure 3.17)

NOTE: The following numbers are connections as shown in Figure 3.17.

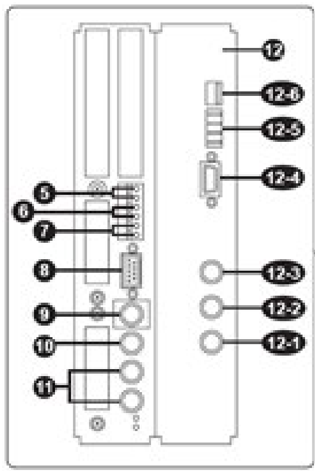


Figure 3.17. Shimadzu GC-2014 Back View Diagram

- 5 START signal input terminal: Connect an external device if any to this terminal to receive a start signal.
- 6 READY signal terminal: Outputs the READY signal to any external device (such as the auto-injector).
- 7 START signal output terminal: Connect an external device if any to this terminal to output the start signal.

All other GC Models

Consult the appropriate GC user manual for instructions on making the remote start cable connections.

Installing the Serial Cable Connections

All GC Models

1. Connect one end of the serial cable (PN 329241, included in the PulseView Monitor software box) to the PC.

2. Connect the other end of the serial cable (PN 329241) to the serial I/O port on the detector controller (Figure 2.4).

Installing the Gas Flow Controls

The gas flow controls associated with the PFPD and the GC can have one of two possible configurations:

- Manual version: The detector controller housing contains the gas flow controls, which operate with manual mass flow controllers on the front of the detector controller.
- EPC-Ready Version: The detector controller housing contains the gas mixing assemblies, and is designed to work with the GC manufacture's EPC or EFC, which supply the gas flows:
  - For the manual version follow instructions below.
  - For the 6890, follow the instructions for the OIM or Aux EPC.
  - For the 7890, follow the instructions for the Aux EPC.
  - For the Shimadzu, follow the instructions for the Aux EPC pressure control manifold.

Follow the relevant method for setting up the configuration for the gas flow controls in your GC.

Gas Flow Controls in the Detector Controller, Manual Version

NOTE: The Detector Controller requires a well-regulated supply of H_2 and air. Pressures in the 50–70 (maximum 90) psi range are acceptable.

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

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. Remove the protective caps from the air inlet and H_2 inlet connectors on the detector controller's back panel.

3. Connect the regulated air supply to the air inlet connector and the regulated H_2 supply to the H_2 inlet connector using the supplied 1/8" nut and ferrule. (Figure 3.18). Push the gas lines into the connector as far as they can go before tightening the nuts onto the connector. Securely tighten the 1/8"nut using a 7/16" wrench.

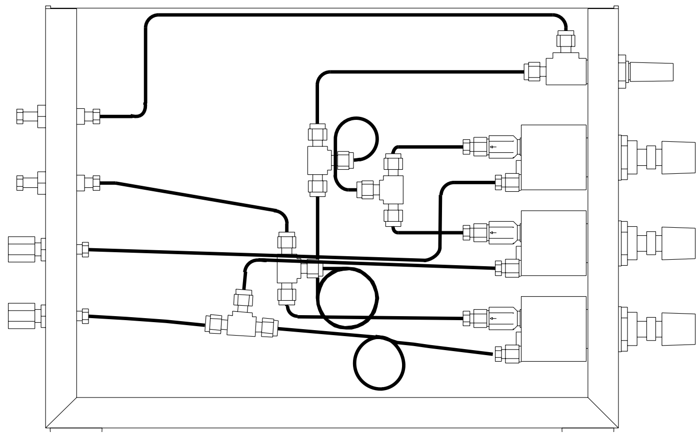


Figure 3.18. Internal View: Manual Gas Flow Controls in the Detector Controller

WARNING: Do not apply supply line pressure to the regulators until it is time to set the gas flows for the PFPD.

4. Route the COMB and WALL gas lines from the PFPD base to the back or side of the GC and out to the back of the detector controller.

5. Remove the plastic protective plugs from the combustor and wall gas connectors and the protective caps on the ends of the combustor and wall gas lines leading from the PFPD base.

6. Connect the combustor gas (COMB) line from the PFPD assembly to the combustor gas connector and the wall gas (WALL) line to the wall gas connector on the back of the detector controller using the attached 1/16" nuts and ferrules. Push the gas lines into the connector as far as they can go before tightening the nuts onto the connector. Securely tighten the nut using a 5/16" wrench.

7. Gently turn the fine adjust needle valve to the off position (clockwise) (Figure 3.18). Do not tighten down as this can damage the needle. Then open the valve four full turns counterclockwise.

8. Set the Air 1 and Air 2 flow controllers as well as the H_2 flow controller to the off position (fully clockwise) (Figure 3.18).

9. Replace the flame arrestor on top of the detector cap with the barbed fitting (PN 202077) included in the PFPD startup kit. Attach a flow meter to the barbed end of the fitting.

10. Ensure the column inlet to the detector is plugged with a no-holed ferrule or that the column is attached (see Installing the GC Column with the column positioning tool).

11. Turn on the air and H_2 gas supplies.

12. Refer to Figure 3.3 Flow Controller Settings.

Installation of the GC EPC Module

Agilent 7890 GC with OIM Pressure Control Manifold

These instructions are specific to the installation of the OI Analytical 5383 PFPD onto an Agilent 7890 GC using the OIM EPC module.

NOTE: As new firmware changes for the GC are released by Agilent, slight changes to this procedure may be required. Please contact OI Analytical Technical Support at (800) 336-1911 for any questions regarding these instructions.

Main Components

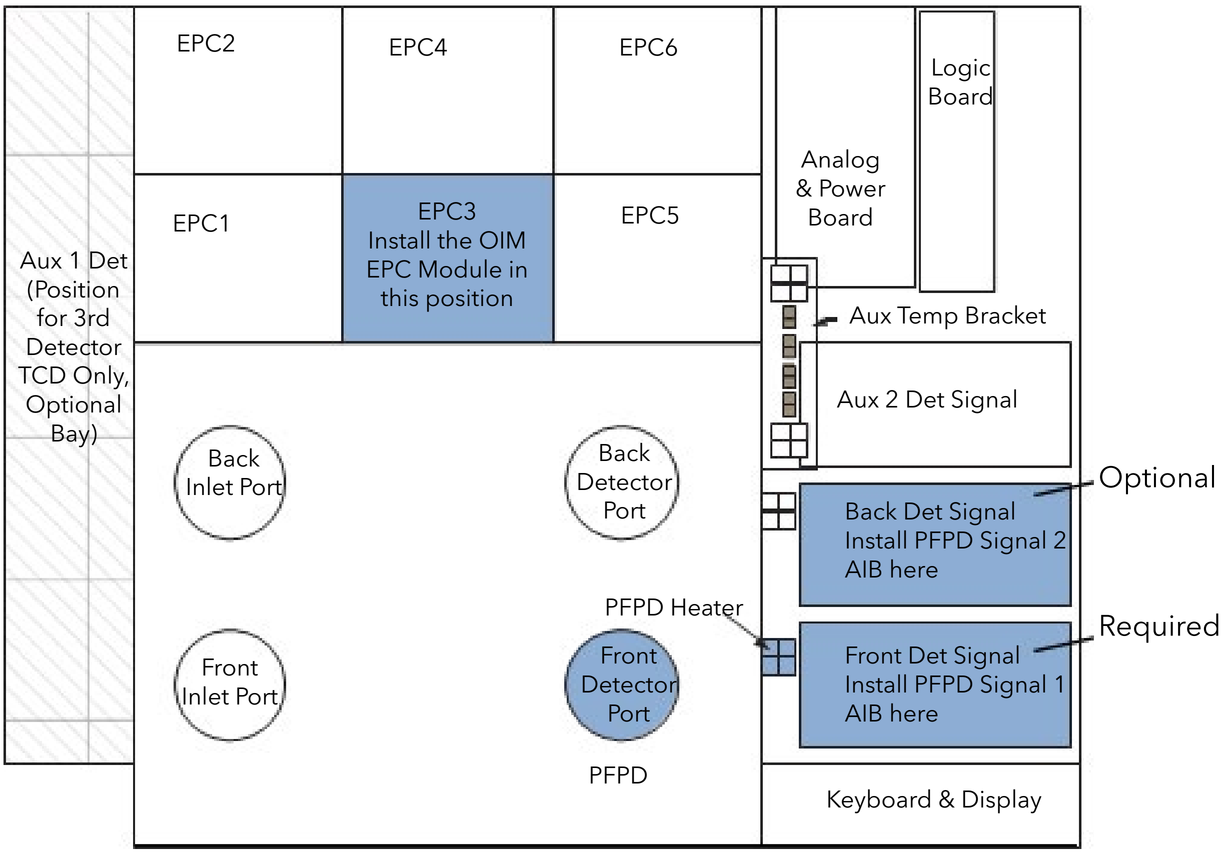
- PFPD detector assembly
- PFPD controller with EPC-ready pneumatics
- 7890 OIM EPC module
- AIB board(s), *1 required*

Main Steps Required for Installation

1. Verify that Agilent 7890 GC firmware revision A.01.05 or later is programmed on the GC.

2. Select the appropriate detector port and flow module position; refer to Figure 3.19 for the normal location or Figure 3.20 for an alternate location.

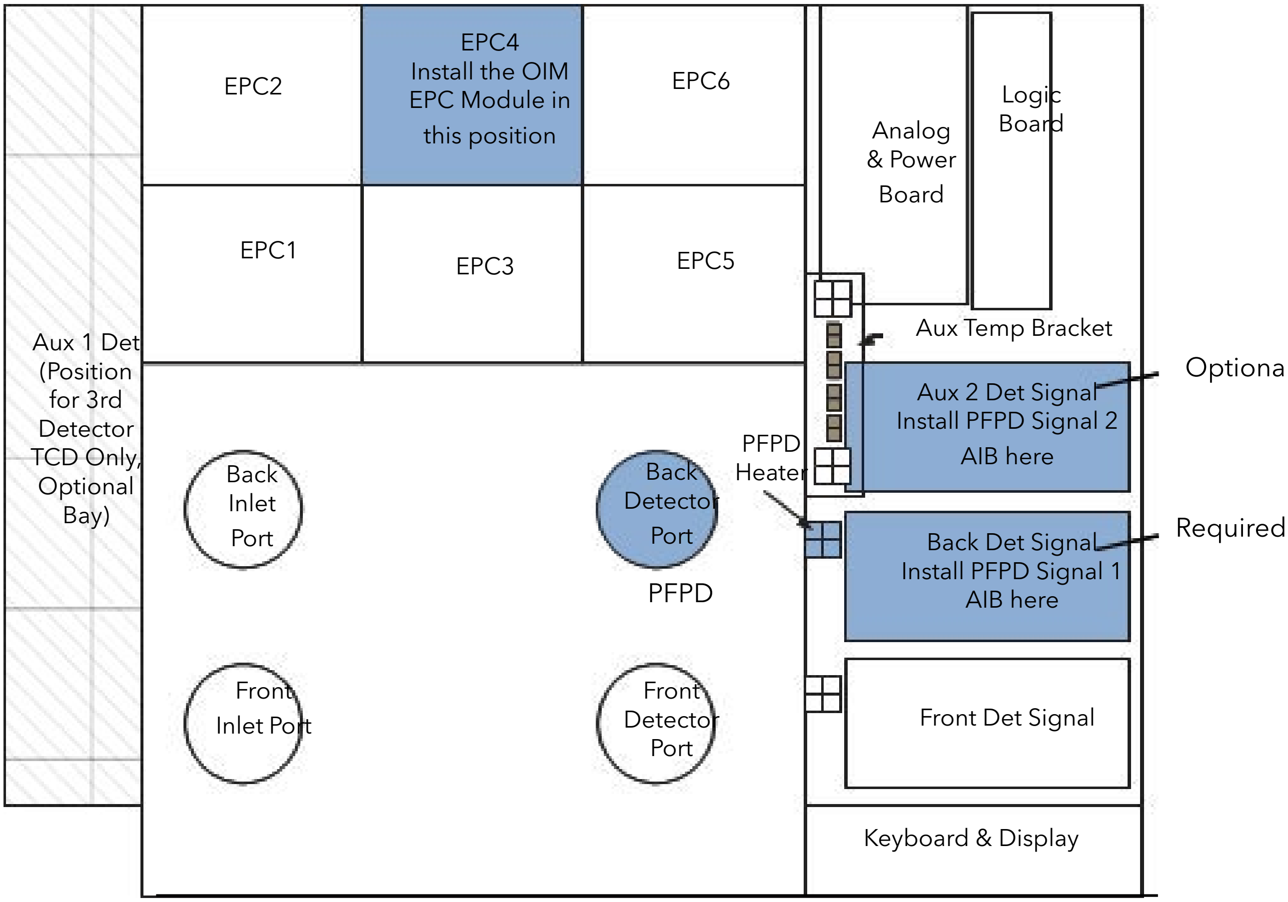
7890 GC Top View *Back of 7890 GC*



Front of 7890 GC

Figure 3.19. Suggested Location for PFPD Components Using the Front Detector Position

7890 GC Top View *Back of 7890 GC*



Front of 7890 GC

Figure 3.20. Suggested Location for PFPD Components Using the Back Detector Position.

3. Install the PFPD assembly in the appropriate detector port as described in the Model 5383 PFPD Operator's manual.

4. Install the PFPD detector controller and cables as directed.

5. Install the OIM EPC module in the 7890 GC.
   a. Remove the EPC module bracket by loosening the captive screws and lifting off the brackets (Figure 3.21).

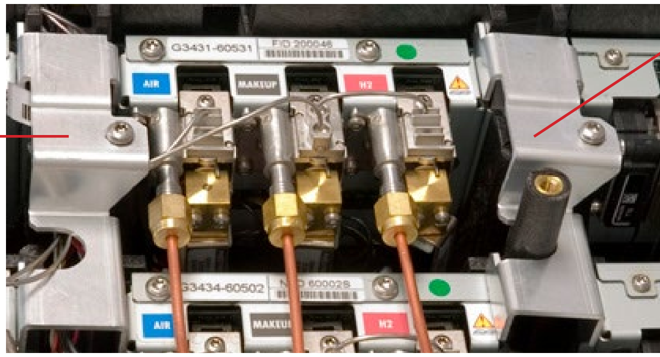


Figure 3.21. Locating the EPC Module Brackets

b. Locate the correct mounting slot for the EPC module (Figures 3.19 and 3.22).

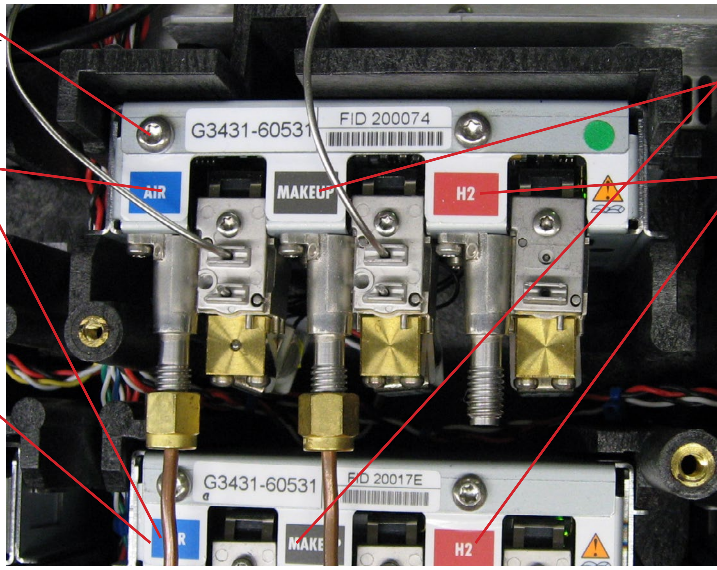


Figure 3.22. Front and Back Detector Positions

c. Attach the communication cable to the EPC module (Figure 3.23).

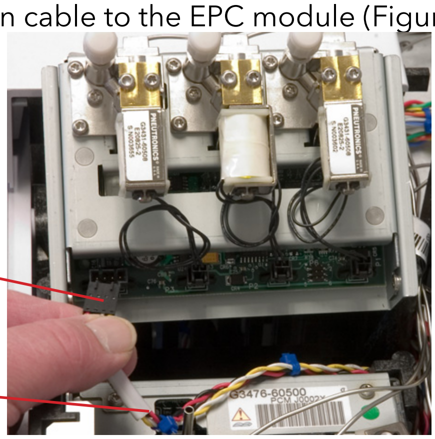


Figure 3.23. Attaching the Communication Cable to the EPC Module

d. Slide the module into the vertical slot tracks. Arrange the cable to prevent it from being pinched by the EPC module (Figure 3.24).

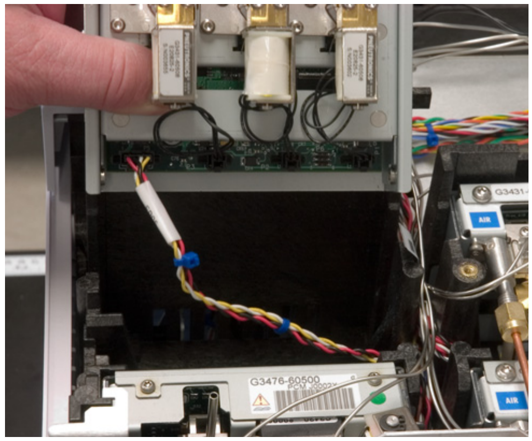


Figure 3.24. Installing the EPC Module

e. Remove the protective caps from the gas inlets.

f. Connect the appropriate gas supplies to the inlets using 1/8" brass nuts and ferrules.

g. Remove the appropriate tubing connector blanks (Figure 3.25) from the EPC module to expose the O-rings and restrictors.

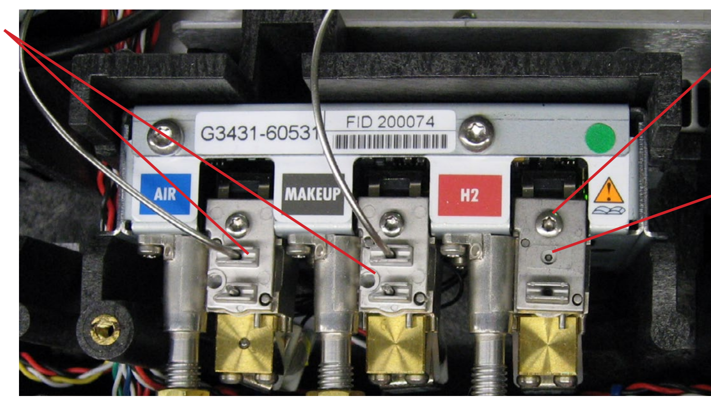


Figure 3.25. Tubing Connector Blanks

h. Attach the tubing blocks (hoodlum block assembly with integrated 1/8" stainless steel lines) to the appropriate manifolds and tighten each screw.

i. Cut off the 1/8" welded nibs on the end of the lines.

j. 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.

k. Connect the gases (see Figure 3.26).
l. Reattach the EPC module brackets using the previously removed screws.

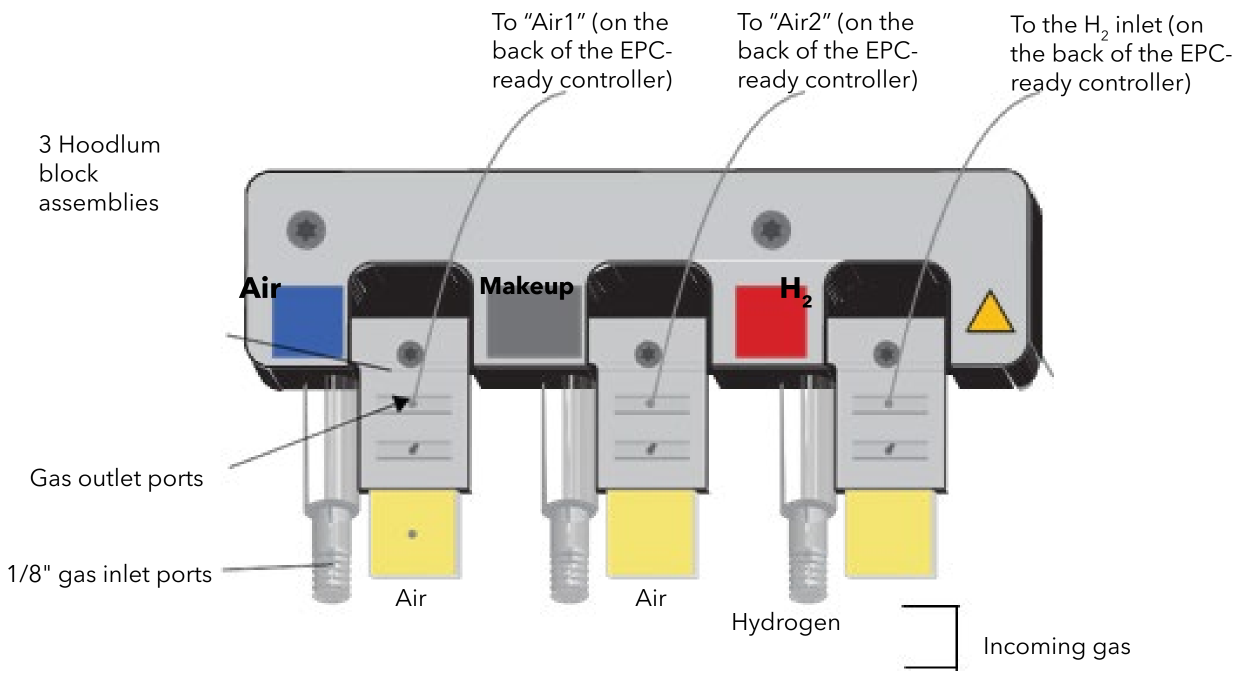


Figure 3.26. OIM Module Plumbing

6. Install the AIB board(s) per the Agilent instructions (see Figure 3.19 for placement).

7. Configure according to Configure the GC.

Configure the 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 Detector

NOTE: The following configuration is for a PFPD installed in the front detector position (as shown in Figure 3.19).

1. Press **Config** and then **Front Det**.

2. Press **Mode/Type**.

3. Scroll to **CPDET AIB, 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** to check the configuration

Configure the Second AIB Board (If Installed)

1. Determine where the AIB board(s) was installed using Figure 3.19.

2. Press **Config** and then the appropriate detector button (either **Back Det** or **Aux Det #**).
   a. For **Back Det**, press **Mode/Type** and scroll to **CPDET AIB, No Htr, No EPC**. Press **Enter**.
   b. For **Aux Det #**, scroll to select **Aux Det 2**.
   c. Press **Mode/Type**. Press **Enter** to select **Install Detector (AIB)**.

3. 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 **Front Det** to view the detector status. Confirm that the detector conditions, detector signal, and three gases display as you scroll through.

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

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

5. Verify the AIB signal displays.

Agilent 7890 GC with Aux EPC Module

These instructions are specific to the installation of the OI Analytical 5383 PFPD onto an Agilent 7890 GC using the Aux EPC module with auxiliary heater control. Refer to the appropriate sections in the PFPD Operator's Manual for additional information on installing the PFPD onto the GC.

NOTE: As new firmware changes for the GC are released by Agilent, slight changes to this procedure may be required. Please contact OI Analytical Technical Support at (800) 336-1911 for any questions regarding these instructions.

Main Components

- PFPD detector assembly
- PFPD controller with EPC-ready pneumatics
- 7890 Aux EPC module and Aux temperature zone kit (containing a bracket, cable, and 2 screws), PN 324543
- AIB board(s), optional

Main Steps Required for Installation

1. Verify that Agilent 7890 GC firmware revision A.01.04 or later is programmed on the GC.
2. Select the appropriate detector port and flow module position; refer to Figure 3.27.
3. Install the PFPD assembly in the appropriate detector port as described in the Model 5383 PFPD Operator's manual.
4. Install the Aux EPC bracket and heater cable as described in the Model 5383 PFPD Operator's manual.
5. Install the PFPD detector controller and cables as directed.
6. Change the restrictors in the 7890 Aux EPC module as described in the Installation chapter of the Model 5383 PFPD Operator's manual.
7. Install the Aux EPC module in the 7890 GC; follow the instructions in the Installation chapter of the Model 5383 PFPD Operator's manual.

NOTE: See Figure 3.27 for recommended installation positions.

8. Connect the gases.
9. Install the AIB board(s) per the Agilent instructions (see Figure 3.27 for placement).
10. Configure according to Configure the GC.

7890 GC Top View

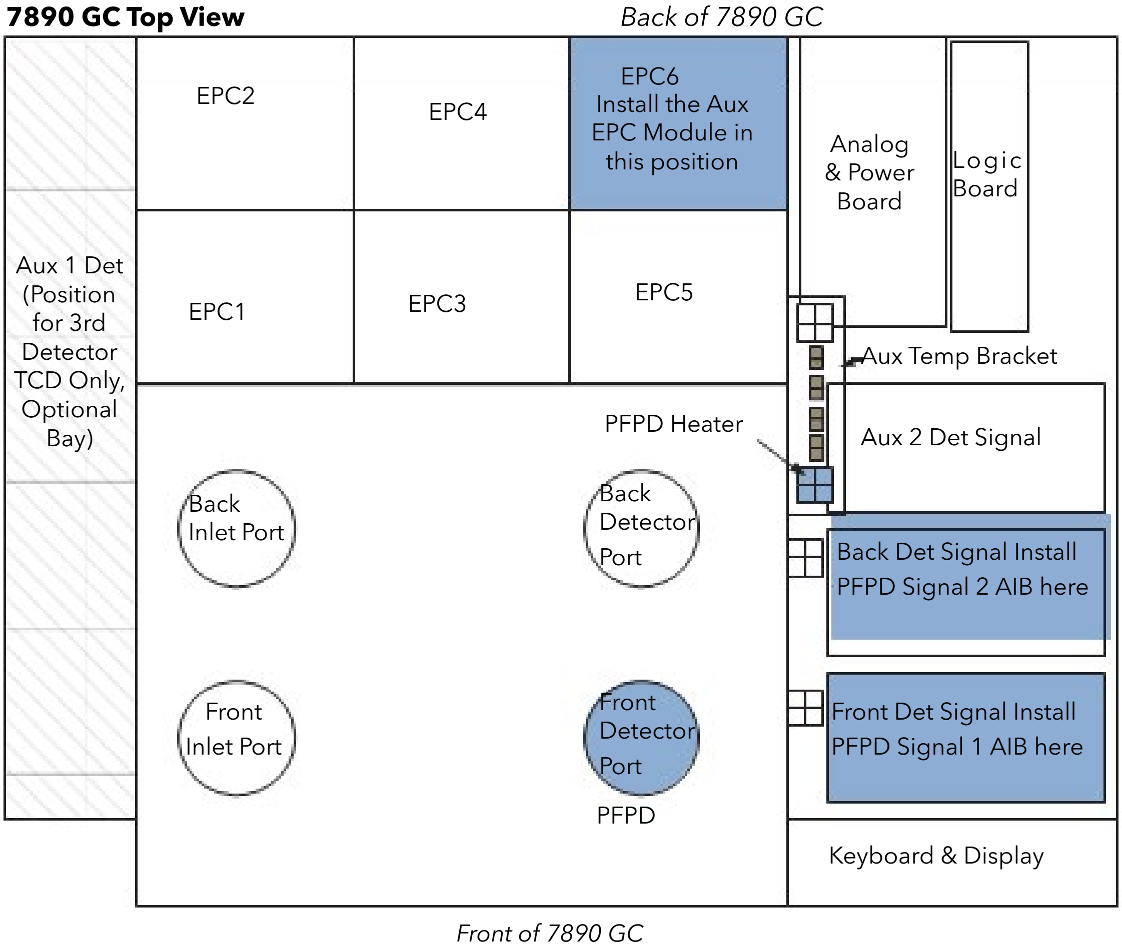


Figure 3.27. Suggested Location for PFPD Components

Preparing the GC

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

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

NOTE: The auxiliary EPC manifold requires a well-regulated supply of H_2 and air.

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

   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 three tubing connector blanks (Figure 3.28) from the Aux EPC module to expose the O-rings and restrictors.

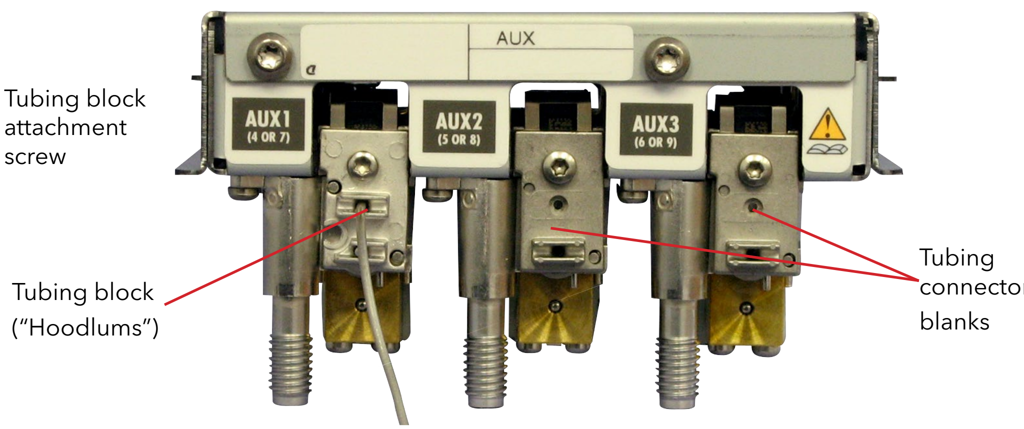


Figure 3.28 Aux EPC Module Tubing Connector Blanks

2. Change to the frits labelled "30 ±1.5 sccm H_2 at 15 psi" on all three channels. 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 three manifolds and tighten each screw (see Figure 3.28).

4. Attach the **H_2** label immediately to the left of the Aux 1 gas connector, the **Air 1** label to the left of the Aux 2 connector, and the **Air 2** label to the left of the Aux 3 connector on the top of the auxiliary pressure control manifold; refer to Figure 3.29.

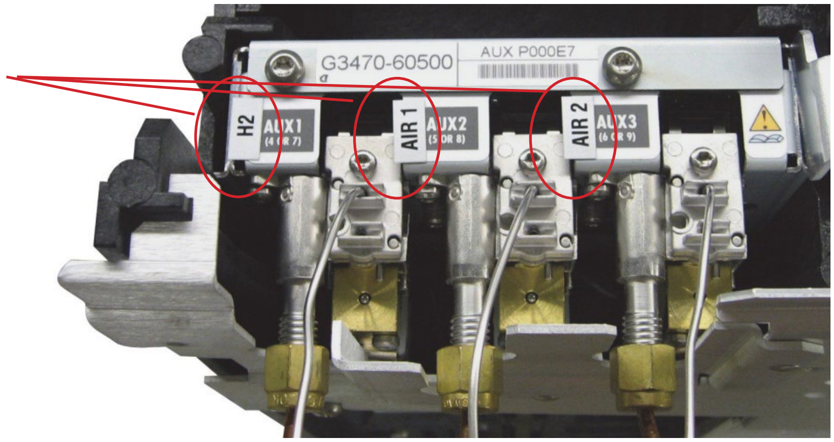


Figure 3.29. Label the Aux EPC

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 3.30); 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 valve
Slot 6	Slot 4	Slot 2	

Back of 7890 GC (gas flow control compartment)

Figure 3.30. Slot Identification

2. Record the inlet channel identification numbers associated with your slot position. These channel numbers are used during the hardware configuration.

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 3.31. 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.

8. Configure the Aux EPC per the "PFPD Installation onto a 7890 GC Using the Aux EPC Module" instructions (PN 324545) provided.

Make the Gas Connections

1. Connect the source gas lines to the manifold using the provided 1/8" nuts and ferrules.

2. Connect the Hydrogen to Aux 1 and the Air to both Aux 2 and Aux 3.

3. Cut off the 1/8" welded nibs on the end of the 1/16" lines.

4. Using the fittings and ferrules supplied in the 5383 startup kit, connect the outlets of the Aux EPC module (1/16" lines) to the back of the 5383 Detector Controller using Table 3.1 as a guide.

Table 3.1. Aux EPC to 5383 Controller Connections

Gas	EPC Channel	5383 Controller
Hydrogen	Aux 1	H_2
Air	Aux 2	Air 1
Air	Aux 3	Air 2

Configure the 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 Aux EPC Module

1. Press **Config** and then **Aux EPC #**.

2. Scroll to the appropriate Aux EPC position and press **Enter**. Refer to Figure 3.27. For example, if the Aux EPC module is installed in the EPC6 position, select **Pressure Aux 1,2,3**.

3. Press **Mode/Type**. Press **Enter** to select the appropriate EPC module. For the previous example, select **Install EPC6**.

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 **Aux EPC #**.

6. 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, Channel 2 is Air 1, and Channel 3 is Air 2.

7. In this case, scroll to **Chan 1 Gas type**. Press **Mode/Type**.

8. Scroll to **Hydrogen** and press **Enter**.

9. Scroll to **Chan 2 Gas type**. Press **Mode/Type**.

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

11. Scroll to **Chan 3 Gas type**. Press **Mode/Type**.

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

Configure the Heater

1. Press **Config** and then **Aux Temp #**.

2. Select the appropriate Thermal Aux position (see Figure 3.27). For example, if the heater is installed in the front Aux position, select **Thermal Aux 1**.

3. Press **Mode/Type**.

4. Press **Enter** to select the appropriate Aux Temp. For the previous example, select **Install Heater Aux 1**.

Configure the AIB Board(s)

1. Determine where the AIB board(s) was installed using Figure 3.27.

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**. Press **Enter** to select **Install Detector (AIB)**.

4. Repeat for each additional AIB card.

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 #, 1** to view the Hydrogen channel. Confirm that the Aux Pressure reading displays for this channel. Repeat for the two additional channels, Air 1 and Air 2.

3. Press **Aux Temp #**. Verify that Thermal Auxiliary 1 appears and that the actual temperature displays.

4. Note where the AIB card(s) are installed. Press the 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.

5. Verify the AIB signal displays.

Agilent 6890 GC with OIM Pressure Control Manifold

The Open Interface Module (OIM) pressure control manifold is the gas flow control device between the main gas source and the PFPD. The preferred gas supply option for the Model 5383 PFPD on an Agilent 6890 is the OIM pressure control manifold (PN 285049), which is mounted in the back of the Agilent 6890 in one of the carrier ports of the gas flow controls marked "Front Detector" or "Back Detector." OI Analytical programs the OIM pressure control manifold for the desired detector configuration before shipping. Alternatively, supply gas through an Aux pressure control manifold (PN 275974), which mounts in the carrier port of the gas flow controls marked "AUX." See the *Agilent 6890 GC Auxiliary Pressure Manifold Installation* for further information.

NOTE: The OIM pressure control manifold requires well-regulated supplies of all required gases to operate the PFPD. Pressures in the 50–70 (maximum 90) psi range are acceptable.

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

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

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

Use the following procedure to install the OIM pressure control manifold into the Agilent 6890.

1. Ensure all gas supply lines used for the PFPD are shut off. Remove the top-back panel on the GC.

2. Remove the protective caps from the gas inlets.

3. Slip the label tag through the bottom slot of the pressure control manifold mounting bracket. Align the mounting bracket over the OIM pressure control manifold.

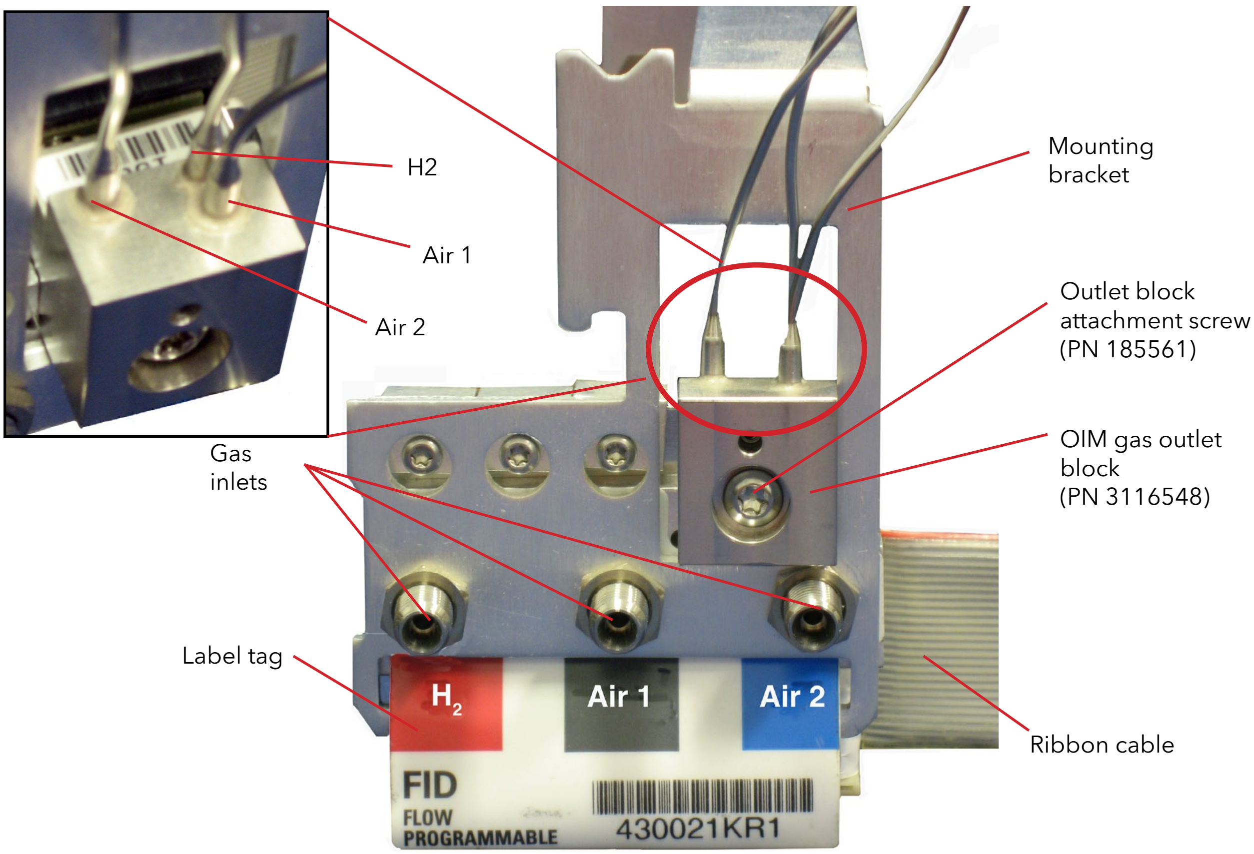


Figure 3.32. OIM Pressure Control Manifold Configured for the PFPD

CAUTION: Do not use the needle valve as an on-off valve. Overtightening can distort the needle valve's seal and make it unreliable.

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

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

6. Route the ribbon cable behind the OIM pressure control manifold.

7. Slide the OIM pressure control manifold into the appropriate "Front Detector" or "Back Detector" slot on the back of the GC until the bracket seats flush with the carrier rails.

8. 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 the ribbon cable is positioned away from the valves and is not pressed against the OIM pressure control manifold.

9. Attach the labels provided to the label tag on the OIM pressure control manifold.

10. Place the labels over the appropriate existing labels: AIR 1 over AIR, AIR 2 over MAKEUP, and H2 over H2.

11. Using needle-nosed cutters, remove the appropriate detector cutout from the top-back panel of the GC

12. Replace the top-back panel on the GC.

13. Remove the protective caps from the gas inlets on the front of the OIM pressure control manifold.

14. Using 1/8" brass nuts and ferrules, attach the H_2 gas line to the inlet labeled "H2", an air line to the inlet labeled "Air 1", and another air line to the inlet labeled "Air 2" (Figure 3.32).

15. Gently turn the fine adjust needle valve (PN 282491) to the off position (clockwise) (Figure 3.18). Do not excessively tighten down, as this can damage the needle valve. Then open four full turns counterclockwise.

16. Proceed to "Gas Connections to the PFPD Detector Controller".

Agilent 6890 GC with Aux EPC Manifold

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

The auxiliary EPC manifold controls the gas flow to the PFPD. It mounts in the back of the Agilent 6890 in the carrier port of the gas flow controls marked "AUX." Use the following procedure to attach the PFPD's gas flow controls to the auxiliary pressure control manifold. Use this configuration as an alternative to the OIM pressure control manifold. This configuration is often used when installing the PFPD into the valve box area of the Agilent 6890.

NOTE: The auxiliary EPC manifold requires a well-regulated supply of H_2 and air. Pressures in the 50–70 (maximum 90) psi range are acceptable.

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

2. Remove the Agilent gas outlet block attached to the front upper right corner of the auxiliary EPC manifold (Figure 3.33) by unscrewing the Torx T-20 screw (PN 185561). Carefully remove the three 30-mL/min cylindrical restrictors (marked with a red dot) and O-rings from the back of the Agilent gas outlet block.

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

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

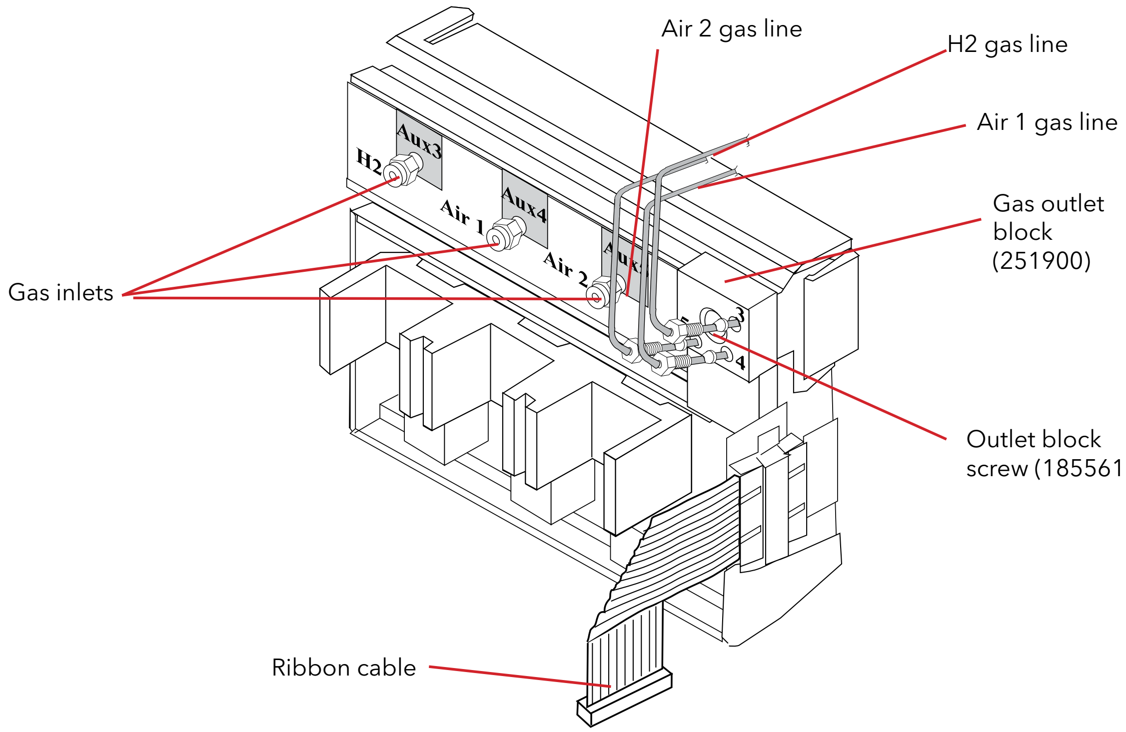


Figure 3.33. Auxiliary EPC Manifold and Aux EPC Mixing Assembly

3. Place two of the Agilent 30-mL/min restrictors and O-rings in the number 4 and 5 gas outlets on the OI gas outlet block (PN 251900). The gas outlet numbers are inscribed on the front of the gas outlet block (Figure 3.33). Place the third O-ring over the supplied 10-mL/min restrictor (PN 290601) and insert the restrictor into the number 3 gas outlet of the OI gas outlet block. With all three restrictors and O-rings in place, reattach the Agilent gas outlet to the OI gas outlet block with the previously removed T-20 Torx screw.

4. Attach the H2 label immediately to the left of the Aux 3 gas connector, the Air 1 label to the left of the Aux 4 connector, and the Air 2 label to the left of the Aux 5 connector on the front of the auxiliary pressure control manifold (Figure 3.33).

5. Locate the troughs on the top and bottom of the auxiliary EPC manifold connector block and the ridges in the carrier compartment of the gas flow controls. Slide the auxiliary EPC manifold into the carrier compartment, lining up the ridges and troughs. From the front of the GC, secure the auxiliary EPC manifold in place by tightening the captive Torx T-20 screw.

6. Plug the auxiliary EPC manifold's ribbon cable into the auxiliary connector facing upward on the electronics board, and push it until the lug is firmly in place. Lock the connector by moving the tabs to the center of the connector until they click into place. (See the *Agilent 6890 GC Auxiliary Pressure Control Manifold Installation Guide* for further instruction.)

7. Remove the protective caps from the Aux 3 (H_2), Aux 4 (Air 1), and Aux 5 (Air 2) gas inlet connectors on the front of the auxiliary EPC manifold (Figure 3.33).

8. Connect the regulated H_2 gas supply to the Aux 3 connector using a 1/8" nut and ferrule.

9. Split the air supply using a tee-connector and attach one arm of the split supply to the Aux 4 connector and the other arm to the Aux 5 connector using a 1/8" nut and ferrule. Push the gas lines into the connector as far as they can go before tightening the nuts onto the connectors. Securely tighten the 1/8" nuts using a 7/16" wrench.

10. Cut the NIBS off the ends of the 1/16"gas lines (H_2, Air 1, and Air 2).

11. Proceed to "Gas Connections to the PFPD Detector Controller".

Shimadzu GC-2014 with Aux APC Module

CAUTION: The maximum pressure into the detector controller must not exceed 100 psi.

The auxiliary pressure control (Aux APC) module controls the gas flow to the PFPD. It mounts in the bottom rear of the Shimadzu GC-2014. Use the following procedure to attach the PFPD's gas flow controls to the auxiliary pressure control module.

NOTE: The Aux APC module requires a well-regulated supply of H_2 and air. Pressures in the 50–70 (maximum 100) psi range are acceptable.

1. Install the Aux APC module according to the instructions supplied with the GC.

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. Route the 2–mm outlet lines from the Aux APC to the back of the PFPD Controller.

3. Attach the **H2** label immediately to the right of the top Aux APC gas connector, the **Air1** label to the right of the middle connector, and the **Air2** label to the right of the bottom connector on the back of the GC at the Aux APC connections (Figure 3.34).

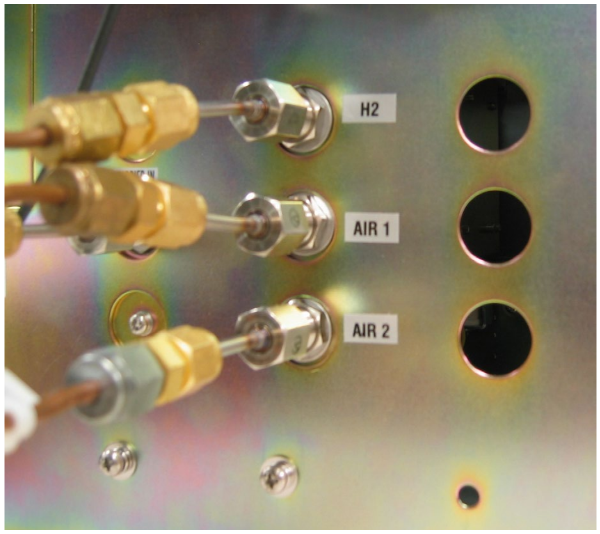


Figure 3.34. Label the Aux APC Gas Connectors

4. Connect the regulated H_2 gas supply to the top position.

5. Split the air supply using a tee-connector:
   a. Attach one arm of the split supply to the Air1 connector.
   b. Attach the other arm to the Air2 connector.

6. Connect the three 2-mm O.D. lines from the Aux APC module to the 2–mm to 1/16 in the startup kit (PN 324342) fitting unions. The 2-mm ferrules should be pre-installed into the union. Push the gas line into the connector as far as it can go before tightening the nut onto the connector. Securely tighten the 2-mm nut using two 12-mm or 1/2" wrenches.

7. Connect the 1/16" tubing from the startup kit (PN 324342) to the other side of the fitting union. The 1/16" ferrules should be preinstalled.

8. To access the Aux APC, press the **Option** button on the GC's front panel. Refer to the Shimadzu GC-2014 manual for instructions on operating the keypad and adjusting the Aux APC flow rates. Refer to the Figure 3.3 Flow Controller Settings.

9. Using the GC's front panel (or software control), turn the H_2, Air1, and Air2 flow rates to zero. Confirm that there is no flow.

10. Customize the Aux APC gas names and heater names by pressing the **FUNC** key on the GC's keypad.
    a. Select **6 GC Configuration**.
    b. Select **6 Port Name Customization**.
    c. Select **3 Aux APC Name Customization**.
    d. Use Table 4.2 to set the names appropriately; use the GC keypad and press **Enter** after each entry.

Table 4.2. Port Names

Port Name	Descriptive Gas Name
APC1	H_2
APC2	AIR1
APC3	AIR2

    e. Return to the previous screen.
    f. Select **1 Heater Name Customization.** DET2 should be listed as type "CON" with the name "CON1".
    g. Rename CON1 to "PFPD" using the keypad. Press **Enter**.

Setting Up the Analytical Line on the Shimadzu GC-2014

An "analytical line" is defined as a method of sample introduction (Shimadzu only), an injection port (with heat control), a column (with heat control), and one or more detectors (with heat control). If not equipped with this line, the GC does not work properly.

For example, the Shimadzu FID electrometer has its own ID. The GC-2014 and GC-2010 expect that when an FID detector with the appropriate resistor in the heater assembly is installed in the GC, the correct electrometer is installed as well. If not, the GC reports a heater or detector error, "No Detector Installed."

To set up the analytical line, perform the following steps.

1. Verify all instructions and precautions have been followed per the Shimadzu GC-2014 Operating Manual. Verify the cables have been installed correctly according to the instructions in this document.

2. Turn on power to the GC.

3. From the front keypad, press **FUNC** (Function).

4. Select menu item **6 GC Configuration**.

5. Select submenu item **3 Transmission Parameter**.

6. Ensure the parameters are set to "Level 3" and "Baud Rate 115200". Press **Apply**.

7. Press **Function**, and select menu item **7 Service/Maintenance**.

8. Select submenu item **1 Installation Position**.

9. Verify the injector and detector are both listed and have an associated position number. The OI Analytical Model 5383 PFPD should be listed as "DET2", and the detector type should be "CON".

   If nothing appears under either detector position, turn off the GC, re-seat the resistor in the heater assembly, and reinstall the heater cable. Turn on power to the GC and repeat steps 3–9.

10. After verifying the installation position, return to the Service/Maintenance submenu by pressing **Function** and menu item **7 Service/Maintenance**. Select item **2 Installation Piping**.

11. Verify the injector is configured per the Shimadzu GC instructions.

12. If the A/D card is not being used, proceed to step 13.

    If using one or two A/D cards, scroll to the DET position ("DET#1" or "DET#2", depending upon the physical location of the A/D card); the Cont. Type should be shown as "OTHER" with no associated heater port.

13. After setting all parameters, turn the power to the GC off and then back on.

14. Press **SET** on the front keypad, and go to "Line Configuration" by pressing **PF2**.

15. Place the appropriate injector and detector in the same analytical line, for example, Line1.

16. Press **OPTION**. The detector temperature and signal settings should now be accessible. Select "Next" to scroll through the menu. Set the detector temperature to "220 °C".

17. Press **Start GC**. Verify the detector heats and a signal displays on the screen.

EPC-Ready for all other GC models

For all other GC makes and models, refer to the appropriate GC user manual for instructions regarding installation and configuration of the associated electronic flow control (EFC) or electronic pressure control (EPC) module. The module and configuration chosen must be capable of providing one well-regulated gas flow line of hydrogen, and two independent gas flow lines of air in the flow ranges shown in Table 3.3.

Gas Connections to the PFPD Detector Controller

All GC Models

1. Connect the hydrogen line from the GC EPC module to the back of the 5383 Controller labeled "H2" using a 1/16" nut and ferrule. (Refer to Figure 2.5.)

2. Connect the Air1 line from the GC EPC module to the back of the 5383 Controller labeled "Air1" using a 1/16" nut and ferrule. (Refer to Figure 2.5.)

3. Connect the Air2 line from the GC EPC module to the back of the 5383 Controller labeled "Air2" using a 1/16" nut and ferrule. (Refer to Figure 2.5.)

4. Remove the plastic protective caps on the ends of the combustor (COMB) and wall (WALL) gas lines leading from the base of the PFPD, and the protective plugs from the combustor and wall gas connectors on the back of the EPC-Ready Detector Controller.

5. Connect the combustor gas line from the base of the PFPD assembly to the back of the 5383 Controller marked "COMB" using a 1/16" nut and ferrule. (Refer to Figure 2.5.)

6. Connect the wall gas line from the base of the PFPD assembly to the back of the 5383 Controller marked "WALL" using a 1/16" nut and ferrule. (Refer to Figure 2.5.)

7. Push the gas lines into the connector as far as they can go before tightening the nuts into the connector. Securely tighten the nuts using a 5/16" wrench.

8. Gently turn the fine adjust needle valve (PN 282491) at the front of the 5383 Controller to the off position (clockwise). Do not tighten down as this can damage the needle. Then open four full turns counterclockwise.

9. Replace the flame arrestor located on top of the detector cap with the barbed fitting (PN 202077) included in the PFPD startup kit. Attach a gas flow meter to the barbed end of the fitting.

10. Open the valves regulating the air and H_2 gas supply to the GC EPC module.

11. Using the GC EPC module controls turn the H_2, Air1, and Air2 flow rates to zero. Confirm that there is no flow.

Setting the Gas Flows

All GC Models

1. Verify that all of the gas flows are off using the flow meter. If any flow registers, check if the Air1, Air2, and H_2 flow controllers are turned off.

NOTE: Allow a few minutes for each gas flow to stabilize before adjusting the next gas flow.

2. Set the initial gas flows to operate the PFPD in the following manner. Adjust the flow controllers as follows (Refer to Table 3.3)
   a. Confirm that the needle valve is open four turns (counterclockwise).
   b. Adjust the Air2 flow controller to supply 10 mL/min.
   c. Adjust the Air1 flow controller to supply 10 mL/min.
   d. Adjust the hydrogen flow controller to supply 11.5 mL/minute.
   e. Once these flow rates equilibrate, do not make any further adjustments to the control valve settings until you are ready to optimize the PFPD operating parameters. (See Chapter 5, Operation.)

NOTE: Be sure to include the column carrier gas flow, which should be < 2.0 mL/min when using He.

Table 3.3 Flow Controller Settings

Gas	Flow setting
Column carrier gas (He)	<2.0 mL/min
Air1	10.0 mL/min
Air2	10.0 mL/min
H2	11.5 mL/min
Total flow	31.5 mL/min + carrier gas

3. Detach the flow meter, remove the barbed fitting, and install the flame arrestor into the detector cap.

Preparing the PFPD for Operation

Before using the Model 5383 PFPD, disassemble the PFPD assembly and install the GC column and combustor. Depending on the element analyzed, changing the optical components may also be necessary.

Disassembling the PFPD

CAUTION: Remove the combustor before disassembling or reassembling the detector body from the detector base. Failure to do this breaks the combustor.

CAUTION: The combustor is very sensitive to dust. If the combustor falls off the extractor tool, replace it or clean it by sonicating it in methanol or flushing with hexane.

The PFPD ships without a combustor in place. Install the combustor prior to operation.

1. Using the 7/64 Allen wrench supplied in the PFPD startup kit, remove the two detector cap screws (PN 295287C) from the detector cap (PN 246900) (Figure 3.35).

2. Carefully lift off the detector cap and place it on a clean, lint-free laboratory tissue.

3. Remove and discard the large aluminum washer (PN 288050) loosely attached to either the bottom of the detector cap or the top of the detector body. This washer should be replaced each time the seal is broken.

4. Remove any previously installed combustor (PN 282939C for the 2-mm combustor, PN 282913C for the 3-mm combustor) from within the detector body using the supplied Teflon combustor extraction tool (PN 280719 for the 2-mm combustor, PN 280727 for the 3-mm combustor). Remove the combustor extraction tool from its protective sheath and gently press its conical tip into the combustor top, which is located just below the upper surface of the detector body. Carefully extract the combustor and immediately reinsert the combustor extraction tool and attached combustor into the combustor extraction protective sheath (Figure 3.36).

NOTE: A combustor is not installed prior to shipping.

CAUTION: Never use bare hands to disassemble the PFPD. Never handle tool parts used to remove or replace internal parts of the detector. Always use clean latex gloves. Use the Teflon extractor device or Teflon-coated forceps to remove and replace internal detector. Place detector parts on a clean, lint-free laboratory tissue or in a clean glass beaker, unless otherwise stated.

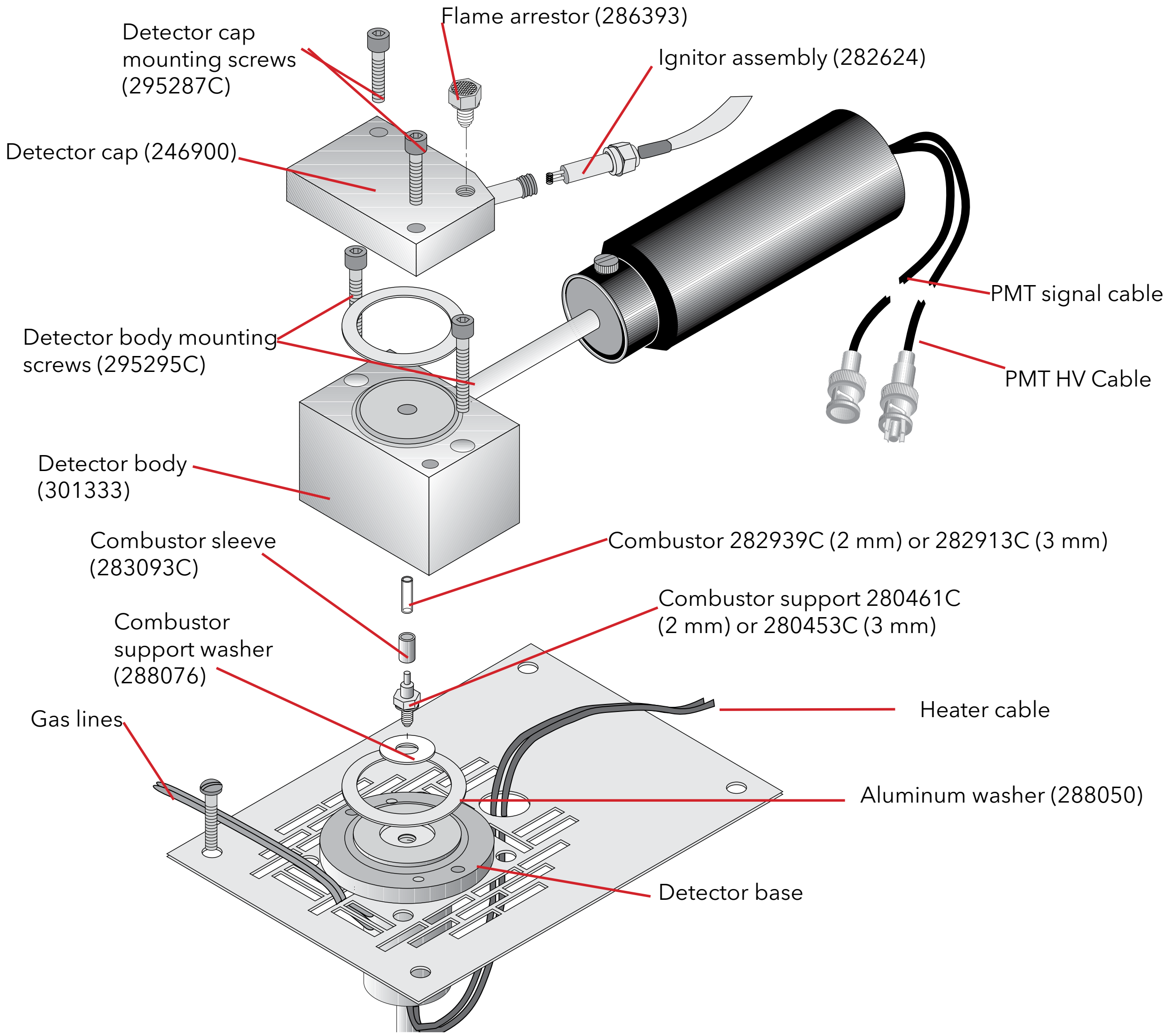


Figure 3.35. Model 5383 PFPD Assembly

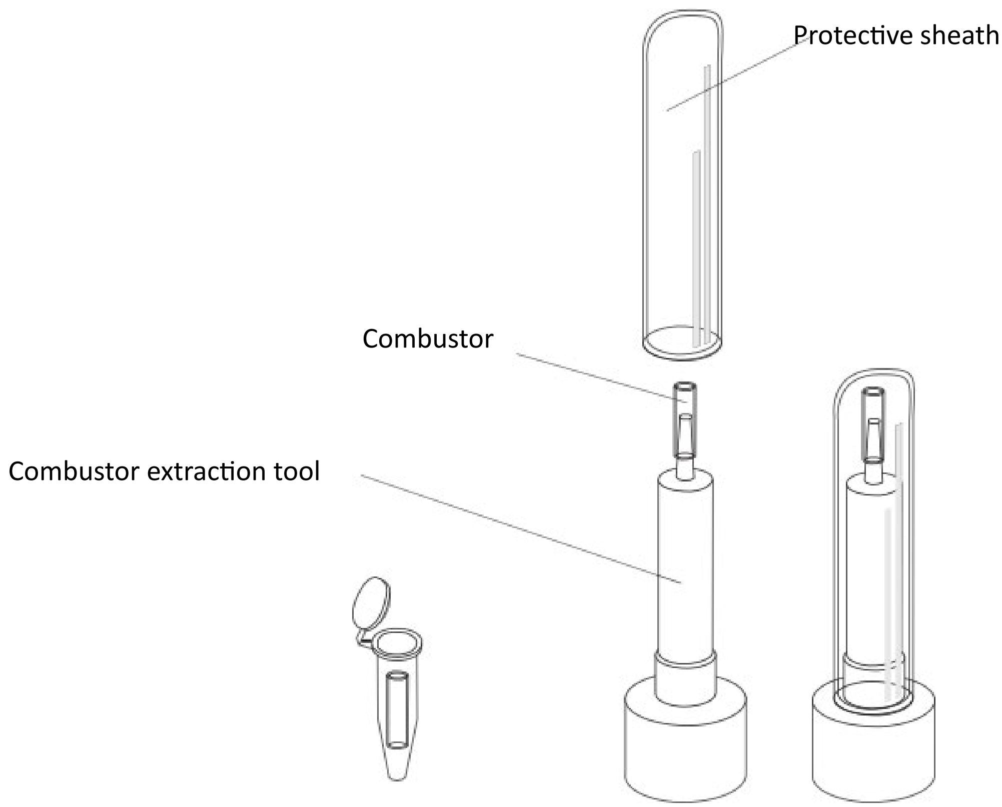


Figure 3.36. Extractor Tool

5. Use the 7/64" Allen wrench to remove the two detector body screws from the detector body (Figure 3.35).

6. Lift off the detector body and place it on a clean, lint-free laboratory tissue.

   Remove and discard the large aluminum washer (PN 288050) loosely attached to either the bottom of the detector body or to the top of the detector base. This washer should be replaced each time the seal is broken.

7. If installing a new combustor, ensure the combustor support corresponds with the combustor. If it does not, refer to Chapter 7 Maintenance, for a description of how to replace the combustor sleeve (PN 283093C), combustor support (PN 280461C for the 2-mm combustor ; PN 280453C for the 3-mm combustor), and the combustor support washer (PN 288076).

Installing the GC Column Without Using the Column Positioning Tool

CAUTION: Crushing the capillary column tip during detector body installation impairs chromatographic performance. Reinstall the column if this occurs.

Reinstall the column whenever changing the combustor support or if operators observe peak tailing, poor peak shape, or poor reproducibility. To minimize degrading the column tip, do not operate the detector at temperatures that exceed the capillary column's recommended maximum temperature. For good chromatographic performance by the PFPD, the end of the capillary column must be square and extend 0.5 mm to 1.5 mm beyond the upper end of the combustor support.

NOTE: Refer to the appropriate GC injection manual for general instructions on installing a capillary column into the injector port.

1. Slide the GC column nut (PN 223057) onto the end of the capillary column. Slide a 1/16" ferrule that matches the outer diameter of the column onto the end of the column with the tapered end facing into the GC column nut (Figure 3.37).

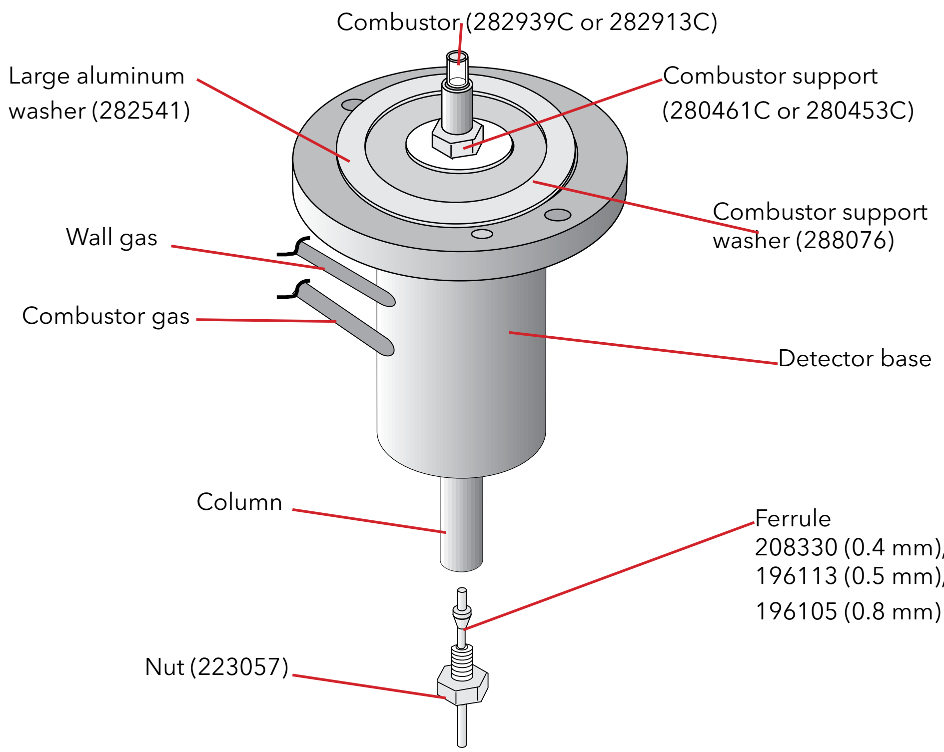


Figure 3.37. Combustor assembly

2. Insert the column end into the detector base with the ferrule and nut on the column. Gently push the column up until it is just beyond the top of the combustor support. Loosely fasten the nut into the end of the detector base.

3. Trim the top of the capillary column using a column scribe. Use a magnifying glass to verify the cut is square cut. Ensure no column or scribe debris falls into the column end.

4. Wipe the column with a clean, methanol-moistened, lint-free laboratory tissue.

5. Carefully pull the capillary column down until the column extends only 0.5 mm to 1.5 mm above the top of the combustor support (Figure 3.38). Tighten the GC column nut to hold the column in position. Using a magnifying glass, verify the top of the column is correctly positioned 0.5 mm to 1.5 mm above the top of the combustor support.

NOTE: For optimal performance, the column must be 0.5–1.5 mm above the combustor support.

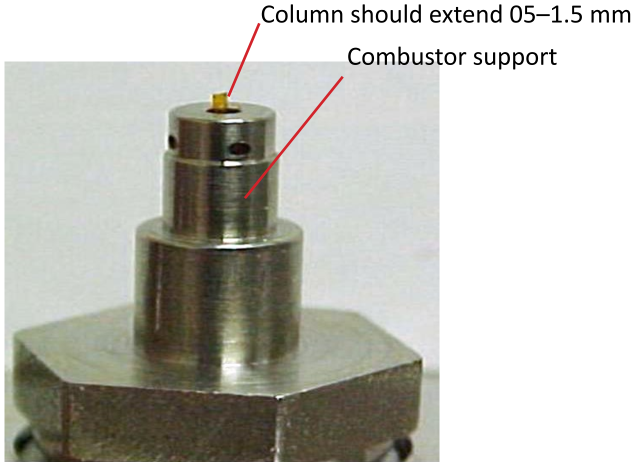


Figure 3.38. Combuster assembly

Installing the GC Column with the Column Positioning Tool

CAUTION: Clean the column positioning tool before inserting it into the detector body. Any dirt or debris on the column positioning tool causes PFPD performance problems.

Use the following instructions for installing the GC column into the Model 5383 PFPD using the PFPD column positioning tool (PN 318949). This tool simplifies column installation and minimizes the possibility of creating performance problems during column installation. The column positioning tool allows proper column installation without removing the detector body.

Reinstall the column whenever changing the combustor support or if operators observe peak tailing, poor peak shape, or poor reproducibility. To minimize degrading the column tip, do not operate the detector at temperatures that exceed the capillary column's recommended maximum temperature. For good chromatographic performance by the PFPD, the end of the capillary column must be square and extend 0.5–1.0 mm beyond the upper end of the combustor support.

NOTE: Refer to the appropriate GC injection manual for general instructions on how to install a capillary column into the injector port.

1. Slide the GC column nut (PN 223057) onto the end of the capillary column. Slide a 1/16" ferrule that matches the outer diameter of the column onto the end of the column with the tapered end facing into the GC column nut (Figure 3.37).

2. With the ferrule and nut on the column, insert the column end into the detector base. Gently push the column up well beyond the top of the detector body so that the column can be cut outside of the detector body. Loosely fasten the column nut into the end of the detector base. Tighten the column nut just enough to have the ferrule gently gripping the column while still allowing column movement.

3. Trim the top of the capillary column using a column scribe. Use a magnifying glass to verify the cut is square cut. Ensure no column or scribe debris falls into the end of the column or the detector body.

4. Wipe the column with a clean, methanol-moistened, lint-free laboratory tissue.

5. Carefully pull the capillary column about 16–19 mm (0.625–0.75") down into the detector body. Ensure the column is below the top of the combustor support before inserting the column positioning tool.

6. Wipe the PFPD column positioning tool with a clean, methanol-moistened, lint-free laboratory tissue.

7. Using a laboratory tissue, **gently** insert the column positioning tool into the detector body (Figure 3.39). The column positioning tool rests on top of the combustor support.

8. Verify the column positioning tool rests on the combustor support by slowly pulling the column down slightly. If the column positioning tool does not move, the column positioning tool is positioned correctly. If the column positioning tool moves, slowly pull the column down until the column positioning tool no longer moves. When the positioning tool stops moving, it rests on the combustor support.

9. The column positioning tool has five grooves that are 0.5 mm (0.020") apart. Use the grooves to judge the proper column placement. Gently move the column up into the detector until the column positioning tool moves up one to two grooves (0.5–1.0 mm).

NOTE: For optimal performance, the column must be 0.5–1.0 mm above the combustor support (Figure 3.38).

10. While holding the column in the proper position, tighten the column nut. The column may move slightly upwards when the column nut is tightened. Observe whether or not the insertion tool moves and adjust accordingly. Moving the tool up 0.5 mm (one groove) is often all that is required. Tightening of the column nut moves the column the remaining distance.

11. Gently remove the column positioning tool. Place it in a clean bag for later use.

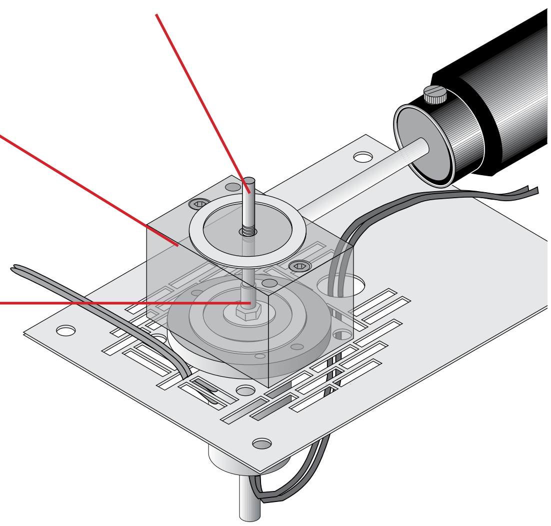


Figure 3.39. Placing the Column Positioning Tool into the Detector Body

Installing the Combustor and Reassembling the PFPD

CAUTION: Do not use Teflon-coated forceps to hold any metallic part. The Teflon coating transfers metallic particles to the combustor, which can scratch or contaminate the combustor surface.

Two sizes of combustors and combustor supports are available. Use a 2-mm combustor for sulfur and selenium detection. Use a 3-mm combustor for detecting other elements. Also, use the 3-mm combustor for sulfur analysis when the column flow rate is above 4 mL/min to reduce sulfur quenching that is caused by a large flux of co-eluting hydrocarbons, or when sulfur is detected simultaneously with other elements that need optimizing.

1. Carefully place the detector body onto the detector base. Use the central hole in the detector body to guide the detector body over the combustor support and sleeve (see the note below on the preferred PFPD orientation).

2. Secure the detector body with the detector body screws (PN 295295C), alternately tightening the screws until the detector body is touching the base.

NOTE: The PMT housing must be as far away from other heated zones (injector, other detectors, etc.) as possible. Route the heater cable away from the PMT and signal cables. Routing alongside the signal or heater cables may contribute to detector noise. Preferred PFPD orientations:

- In the Agilent 6890 and 7890, the preferred PFPD orientation has the PMT housing assembly extending toward the right into the electronics compartment (Figure 3.5). This minimizes interference with an optional valve box that can be mounted in the center of the detector area. However, with this orientation, the right rim of the GC detector cover and the left rim of the electronics top cover must be cut out to fit over the PMT housing assembly. To change the PFPD orientation, remove the detector body from the detector base, reposition the detector body in the desired orientation, and reattach it to the detector base using the detector body mounting screws. Reassemble the rest of the detector as previously described.

- In the Shimadzu GC-2014, the preferred PFPD orientation has the PMT housing assembly extending toward the left. The detector is located in the back of the instrument.

- On all other GC models, mount the PFPD sop that the PMT housing is as far away from other heated zones as possible. Consult the appropriate GC user to manual for guidance.

3. Use the supplied Teflon combustor extraction tool to install the combustor (PN 282939C for the 2-mm combustor, PN 282913C for the 3-mm combustor) into the hole in the detector body. When a new combustor is being installed, use the combustor extraction tool to remove the combustor from its protective container by gently pressing the conical end of the combustor extraction tool into the open end of the combustor.

4. Lower the combustor vertically into the hole in the detector body. If the combustor sticks to the combustor extraction tool, use the supplied Teflon-coated forceps to gently pry the combustor off of the tool and into the hole in the detector body. The combustor is correctly installed when the top of the combustor sits approximately 0.05" (1.25 mm) below the top surface of the detector body.

   Place a new aluminum washer (PN 288050) on the detector body. Use clean metal forceps to place and center the new washer on the center hole in the detector body (Figure 3.35).

5. Install the detector cap (PN 246900) on the detector body top. Adjust the position of the detector cap until it is centered on the sealing washer and the detector body.

6. Secure the detector cap with the detector cap screws (PN 295287C), alternately tightening the two screws. A gap exists between the detector body and the detector cap (approximately 0.5 mm). This gap should be the same all around.

7. If the ignitor assembly cable is not already attached to the detector cap, thread it into the detector cap until it is snug. Center the ignitor coil in the exit vent of the detector cap.

8. Replace all of the previously removed GC covers.

Installing PulseView Software

Before installing the PulseView software, verify the PC meets the minimum hardware and software specifications required to run PulseView (see Chapter 1 Requirements).

Insert the PulseView CD into the appropriate drive on the PC.

NOTE: To install this software, the operator MUST be logged in with Administrator privileges for the PC. Consult with your IT department for assistance.

NOTE: Prior to installing the PulseView software, be sure to perform all Windows updates available for this PC and operating system.

Installing the 5383 PulseView Software and Drivers

Follow these steps to install the PulseView software.

1. Insert the OI 5383 PulseView Software Installation CD into the CD drive on the PC.

NOTE: If the AutoRun feature does not automatically launch the Install program, go to "\<CD Drive>\OI 5383 SW" directory, and right-click **Setup.exe** and select **Run as Administrator**.

2. Click **Next** to begin the installation process (Figure 3.40).

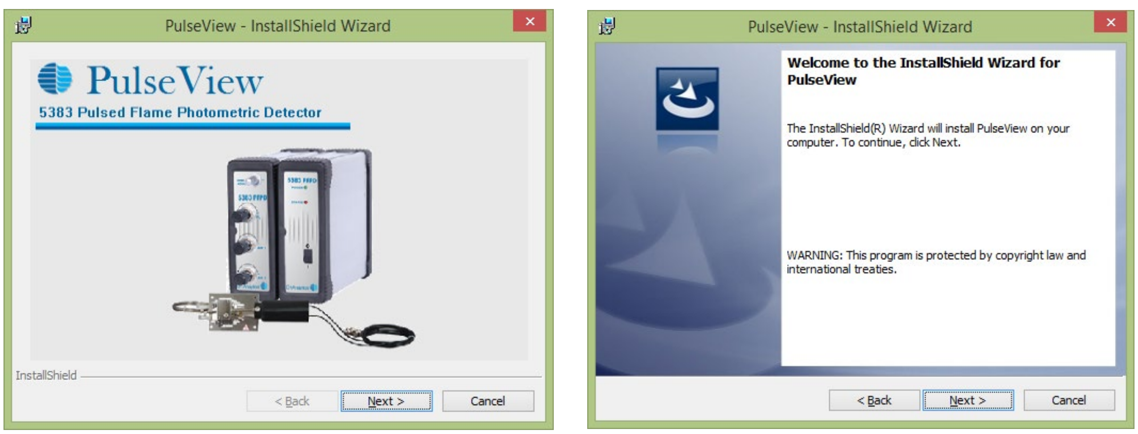


Figure 3.40. PulseView Software Installation

3. Click **Next** to review the installation path and information (Figure 3.41).

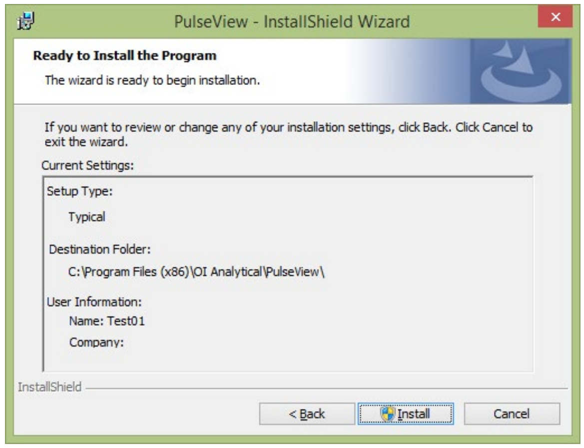


Figure 3.41. PulseView Software Installation Path

4. Click **Install** to begin the installation process and follow the instructions on the screen (Figure 3.42).

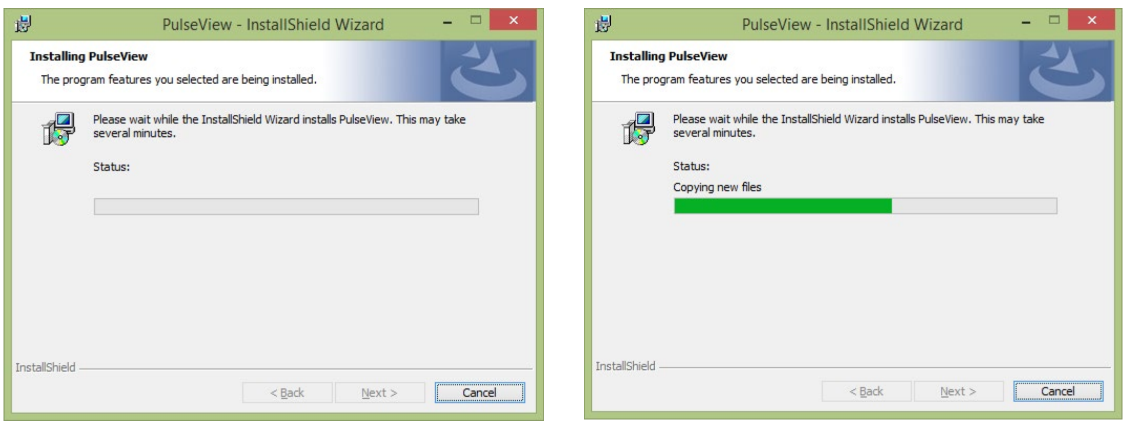


Figure 3.42. PulseView Software Installation in Progress

5. Click **Finish** when the software installation is complete (Figure 3.43).

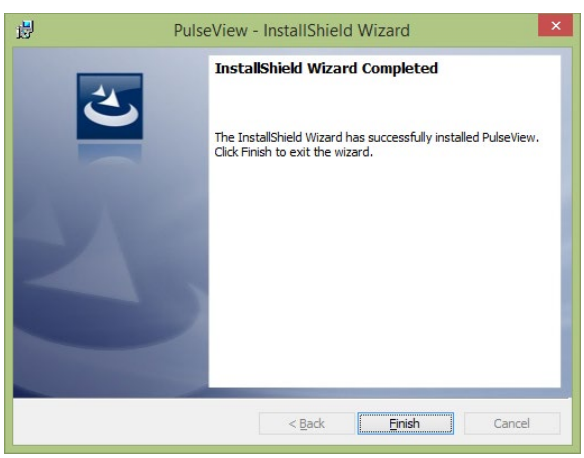


Figure 3.43. PulseView Software Installation Complete

6. Once the 5383 PulseView software is installed, the USB-to-RS-485 Converter FTDI CDM Driver program will automatically begin installation.

7. Click **Extract** to unpack the driver for installation (Figure 3.44).

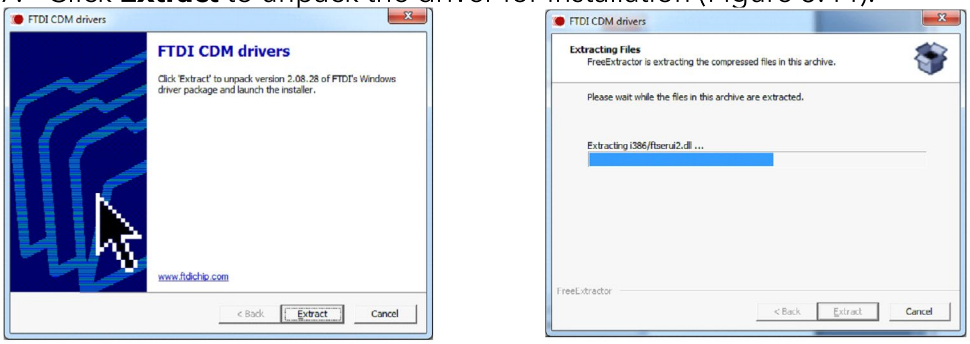


Figure 3.44. FTDI - Begin Installation

8. Click **Next** to begin installation (Figure 3.45).

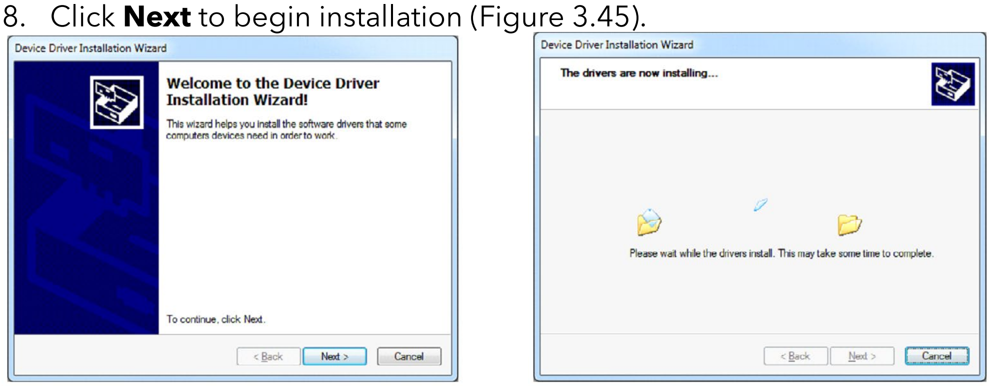


Figure 3.45. FTDI - Installation in Progress

9. Click **Finish** when the device driver installation is complete (Figure 3.46).

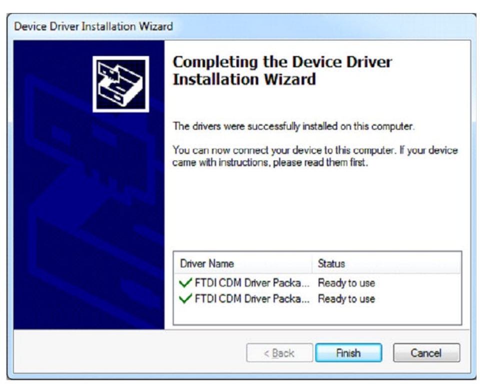


Figure 3.46. FTDI - Installation Complete

10. The 5383 PulseView software is now installed and ready to be configured.

Configuring the PulseView Software Connection

Connecting and Configuring the USB-to-RS-485 Converter

The USB-to-RS-485 Converter is used to connect the PulseView software to the 5383 PFPD system. When the converter is plugged into the USB port, the device driver program will automatically configure the USB port to act as a Virtual Com Port (VCP). That VCP will then be used in the 5383 PulseView Launcher program to configure a launch icon for the 5383 system. (See PulseView Launcher.)

NOTE: If more than one 5383 system is going to be connected to the same PC, then one USB-to-RS-485 Converter and an available USB port will be required for each connection.

Follow these steps to configure the USB-to-RS-485 Converter:

1. Insert the USB end of the USB-to-RS-485 Converter into an available USB port in the PC. Be sure the cable can reach all the way to the 5383 unit.

2. Windows should automatically recognize that the Converter has been installed and run the FTDI driver to assign it a Virtual Com Port (VCP).

3. Verify the assigned VCP by accessing the Control Panel->Device Manager screen and reviewing the Ports section. A new VCP should have been assigned. See Figure 3.47 for COM15.

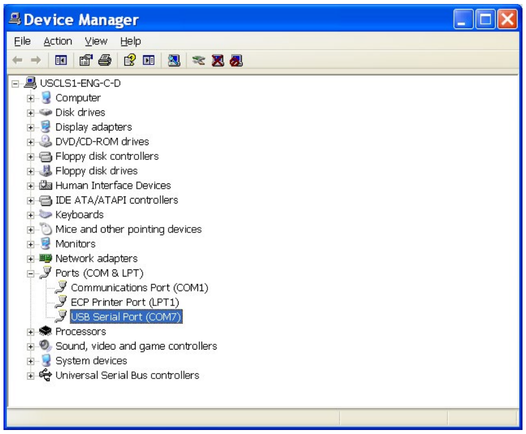


Figure 3.47. Device Manager - Ports

4. This VCP should be used in the 5383 PulseView Launcher application when creating an icon for this 5383 unit.

5. Connect the other end of this cable to the RS-485-to-PC port on the rear of the 5383 Electronics Controller.

PulseView Launcher

The 5383 PulseView Launcher software is used to configure launch icons for each 5383 system, and to provide access to the PulseView Monitor software and the PulseView Analyzer software. After installing the 5383 PulseView software onto a suitable PC, and configuring to the USB-to-RS-485 converter, launch the PulseView Launcher software from the Start menu (Figure 3.48).

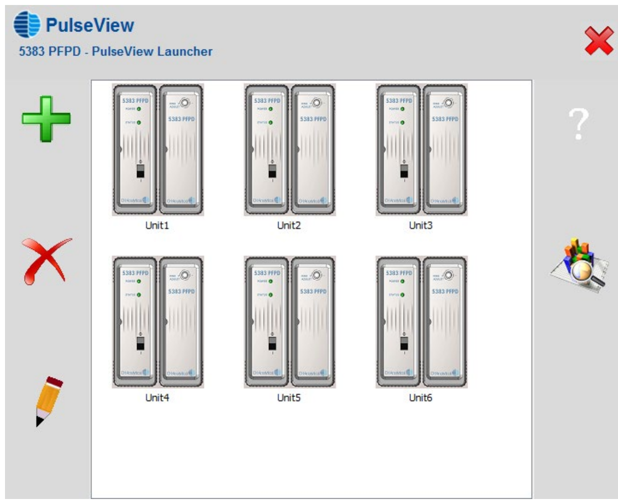


Figure 3.48. PulseView Software Launch Screen

To add a new 5383 system to the launcher list, press the Add (+) button, and give the instrument an appropriate name (typically the instrument serial number) and select a COM port address (as created during the USB-to-RS-485 Converter installation process) (Figure 3.49).

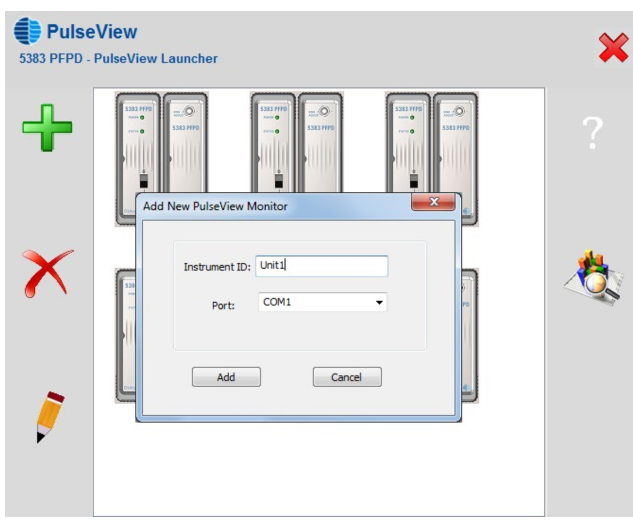


Figure 3.49. PulseView Launcher - Add Instrument

To make changes to a system configuration, select the system icon to be changed and press the **Modify** button, which brings up the Modify screen (Figure 3.50).

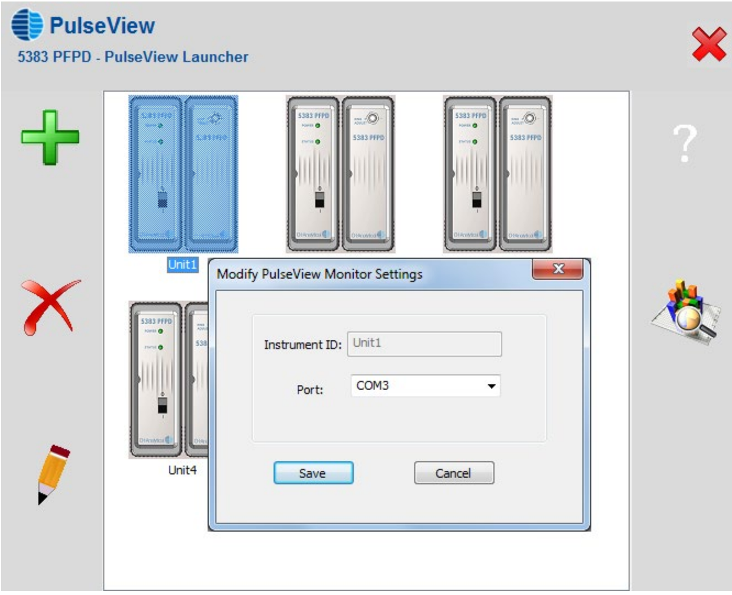


Figure 3.50. PulseView - Modify Instrument

Also available in the launcher are a help file and the PulseView Analyzer. The icons for these features are shown below.

PulseView - Help

PulseView Analylzer 

Getting Offline Help

To get offline help about system functionality or operations, the operator can access the 5383 PulseView Operator's Manual by clicking the Help (?) button on the PulseView Launcher screen. A PDF version of this 5383 Operator's Manual will be automatically displayed.

Chapter 4 PulseView Monitor Software

This chapter describes the features of PulseView Monitor software developed by OI Analytical to set up and optimize all PFPD operating parameters. PulseView Monitor is a Windows-based software that lets the operator easily observe the amplitude and lifetime of chemiluminescent emissions resulting from analyte combustion in the PFPD. The following gives an overview of the Main screen, the button features, and the secondary screens contained in PulseView Monitor.

Starting the PulseView Software

Launching the Software

To launch the 5383 PulseView software, double-click the launch icon of the desired 5383. Once connected to the 5383, the PulseView Home screen will appear.

PulseView Monitor Main Screen

The main screen consists of a menu bar, a real time display of the chemiluminescent signal, and several screen manipulation and data recording features along the right and bottom of the screen (Figure 4.1). The functions of the menu bar elements are described in the section below.

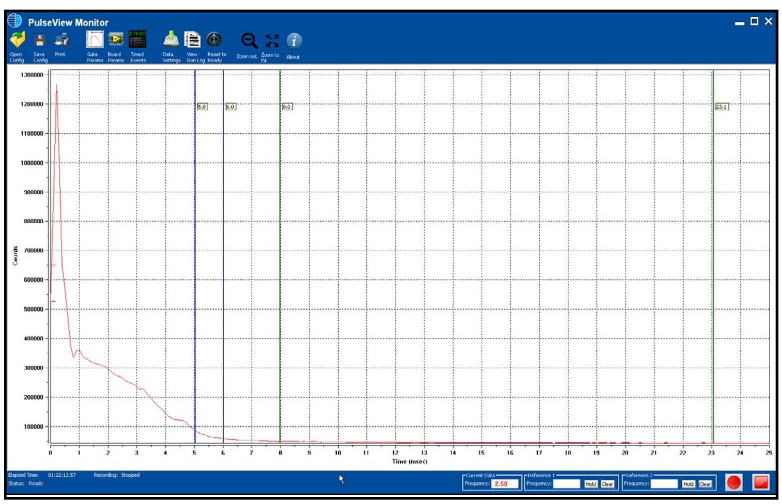


Figure 4.1. PulseView Monitor Main screen

The **title bar** displays the name of the program and the name of the instrument being controlled. It also provides buttons to minimize and maximize the screen size, as well as exit the PulseView Monitor program.

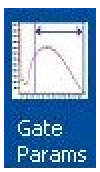
The **button toolbar** at the top of the screen allows the operator select one of several operations or screens, as shown in Table 4.1.

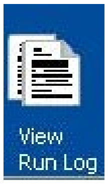
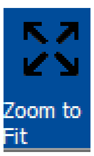
The **real-time graph** continually updates with the latest chemiluminescent signal pulse from the PFPD detector. See more details in the section below.

The **status bar** displays the elapsed time and pulse frequency values, and provides the Start and Stop recording buttons.

Button Bar Features

Table 4.1. Summary of each button on the Button Bar

Button	Purpose
	The Open Configuration screen allows the operator to select which saved configuration file (.cnfg) to open and apply to the 5383 PFPD system. A dialog box appears asking, "Save New PFPD Parameters to EEPROM?" Select **Yes** to update the EEPROM with the new parameters.
	The Save Configuration screen allows the operator to specify a file name and directory location to save the current configuration settings. These can be recalled later using the Open Config button.
	The Print Configuration screen allows the operator to print all of the configuration parameters for the Gates, Board/Channel, and Timed Events screens for record-keeping purposes.
	Accesses the Gate Parameters screen, which is used to set gate values and identify gate mode labels. (For more information, see Figure 4.2 Gate Parameters Screen.)
	Accesses the Board/Channel Parameters screen, which is used to set the parameters for the PFPD Board and the Channel 1 and Channel 2 output signals to the GC. (For more information, see Figure 4.3 Board/Channel Parameters Screen.)
	Accesses the Timed Events screen, which allows the operator to program events to occur based on the time since the start of the run. (For more information, see Figure 4.4 Timed Events Screen.)
	The Data Settings screen allows the operator to specify the filename and indexing parameter used when saving data to the .PFPD file during Record sessions.

	Displays the Run Log screen, which reflects all data recording events (both manual and automatic) with a time and date stamp. Periodically save or delete this log.
	Resets the Detector Controller clock to 0.00 minutes. The clock timer activates by a contact closure supplied by either the GC or a remote start switch.
	Zooms out to the previous zoom level.
	Zooms out to fit the entire pulse on the screen in both the X and Y axes.
	The About screen provides system software and firmware version information. It also provides some real-time feedback of a few status variables.

Real-Time Graph Features

The real-time graph continually updates with the latest Chemiluminescent signal pulse from the PFPD detector. It also displays up to two static "reference" pulses for comparison when changing configuration parameters.

Zoom In	The real-time graph provides the ability to zoom in to view specific graph details, as needed. To zoom in, use the left mouse button to click and drag to define the zoom area. Left-click the mouse in the upper-left area of the desired zoom region, and then drag the mouse to the lower-right area of the region and release the left mouse button. The graph will immediately update to show the new zoomed in display. Up to 10 levels of zoom-in will be stored for use during zoom-out.
Zoom Out	Zoom out in the real-time graph provides the ability to return the graph display to the previous zoom level. This can be done by either clicking the **Zoom Out** button in the button bar, or by a shortcut using **Ctrl + Left-click**. Up to 10 levels of zoom-in can be undone by the zoom-out feature.
Zoom to Fit	To zoom the real-time graph to best fit the graph data currently being displayed (in both X and Y axes), use the Zoom to Fit option. This can be done by right-clicking the mouse on the Y-Axis, and then selecting the **Zoom to Fit** option. **NOTE:** Zoom in/zoom-out features are independent of the multiplier values set in the Gate Parameters and Timed Events screens. These do not affect the output signals sent to the data integration device.
Reference Markers	PulseView provides two green vertical bars and two blue vertical bars that delineate movable marker pairs. Change the position of the vertical bars associated with each marker pair by dragging them left or right with the cursor. The time value of each marker is displayed and updated as the marker is moved. **NOTE:** These markers are only for reference, and are not tied to any values in the Gate Parameters screen.

Status Bar Features

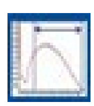
The status bar at the bottom of the screen provides the following information:

- Elapsed Time since the start of the last run
- PulseView Monitor is ready to record PFPD signal data
- PulseView file name is recorded
- Chromatographic run is in progress
- Current, Reference 1, and Reference 2 pulse data
- Start and Stop recording buttons

Elapsed Time	Displays the time elapsed since the start of the last run. The Elapsed time automatically resets to 0 when a GC Start is received or a when a Reset to Ready button is pressed.
Current Data	Displays the PFPD's current pulse frequency in Hz.
Status	Displays as Ready or Running. Ready indicates that the PFPD is ready to start when a GC run is initiated. Running indicates that a GC run is in progress.
Recording	Displays as Stopped or In Progress. Stopped indicates that Monitor is available to begin recording a PFPD file. In Progress indicates that Monitor is actively recording a PFPD file.
Record File	Displays the name of the .PFPD file being recorded (or last recorded if stopped). (See the Data Setting Screen for information on the file naming.)
Reference 1 and 2	Allows the user to set one or two chemiluminescent signals as references by clicking **Hold**. The Reference 1 signal is blue, the Reference 2 signal is green, and the active signal is red. The PFPD pulse frequency when the reference signal was set also appears. Clicking **Clear** for either reference removes that reference signal from the screen.
Start (record)	Provides the ability to Start recording pulse data to the .PFPD file for use by PulseView Analyzer. The filename and path are determined by the values entered in the Data Settings screen. This screen provides the ability to auto-increment the filename numbering scheme, so that successive Starts will automatically create unique filenames.
Stop (record)	Provides the ability to Stop recording pulse data to the PFPD file. This closes the current .PFPD data file so that no further data is appended. This file is now ready for use by PulseView Analyzer.

PulseView Monitor Screens

Gate Parameters Screen

To access the Gate Parameters screen, click  located on the toolbar (Figure 4.2).

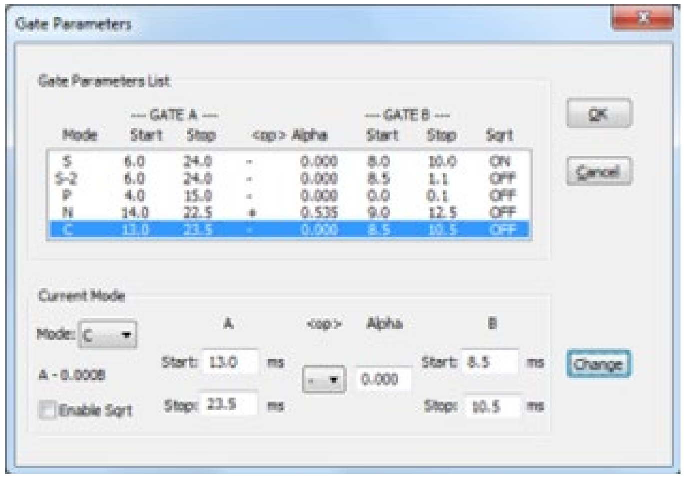


Figure 4.2 Gate Parameters Screen

Use the Gate Parameters screen to select gate mode identification labels and set gate values.

Gate Parameters List Shows the current gate parameters assigned to each specified mode. To modify a mode, highlight the row to be modified in the Gate Parameters List and change the desired parameters.

Start and Stop Specifies the integration gate start and stop times (in milliseconds from the start of each pulse). To maximize PFPD selectivity for the specified mode, set the start and stop times to incorporate as much of the selected mode's emission time domain as possible, and minimize overlap with the emission time domain of other modes detectable with the optical filter used.

<op> Sets an algebraic operator (–, +, x, or /) for the Alpha scaling factor used with the dual-gate subtraction feature.

Alpha Specifies the scaling factor used for the dual gate technique, which is activated when Alpha has a nonzero value. Chapter 14, "Dual Gate Capability and Alpha Calculation describes how to use the dual gate capacity and how to determine appropriate Alpha values.

Enable Sqrt Activates the square root calculation of the output signal used to linearize quadratic response signals associated with some elements (e.g., sulfur mode).

The output signal for a specified mode is a function of the selected operator (<op>), the specified value of the Alpha scaling factor, and the integration results for gates A and B (which are determined by the specified start and stop times). PulseView Monitor calculates the output signal as follows:

Output signal (mode) = A <op> Alpha x B

NOTE: If Alpha is zero, then the output signal is the gate A signal only.

The function of each button along the right side of the Gate Parameters screen follows:

OK	Transmits the current gate parameters to the detector controller and exits the Gate Parameters screen.
Cancel	Disables all changes made to the current gate parameters and exits the screen without downloading changes to the detector controller.
Change	Modifies the current gate parameter without appending a new mode to the gate parameters list. A total of five modes can be specified.

NOTE: Changes are automatically transferred to the 5383 unit and saved to the EEPROM when **OK** is pressed.

Board/Channel Parameters Screen

To access the Board/Channel Parameters screen, click the **Board Params** icon located on the toolbar (Figure 4.3).

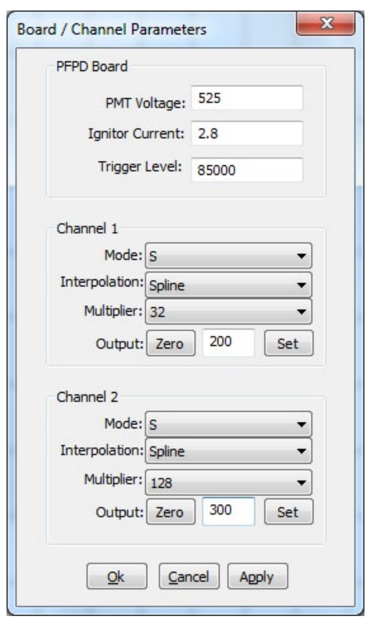


Figure 4.3. Board/Channel Parameters Screen

The Board/Channel Parameters screen sets the parameters for the PFPD Board and the Channel 1 and Channel 2 output signals. The Channel 1 and Channel 2 parameters specify the output signals sent to the data handling device. Two channels are provided because the detector controller can simultaneously process and send two signals. The function of each field in this screen follows:

Board Parameters

PMT voltage	Sets the voltage delivered to the photocathode of the PMT to convert photon energy to an electrical current. The range is 0–1,000 V and the default is 525 V. Increase the PMT's sensitivity to an analyte (represented by chemiluminescent amplitude) by increasing the PMT voltage. Noise usually increases as PMT voltage is raised above some value.
Ignitor Current	Sets the current level for the PFPD ignitor to properly ignite the gases. Range is 0-3.0A. Default is typically 2.8A.
Trigger Level	Sets the trigger level for pulse detection. The trigger level is used to determine when to start acquiring the pulse data. The trigger level should be set at the point where the pulses displayed are the most consistent. The range for the trigger level is 0–8,000,000. The trigger level should be set so that the counts are 50-75% of the combustor/ignition peak (the tallest peak at ~ 0-1 milliseconds).

Channel Parameters

Mode	Assigns one of the current modes in the gate parameters list to the signal output channel.
Interpolation	Sets the method for interpolating between observed output values from each pulse to derive a higher output data frequency. Linear provides point-to-point interpolation. Spline provides piece-wise cubic smooth interpolation.
Multiplier	Sets the integer (1, 2, 4, 8, 16 … 4096) used to multiply the output signal of the associated channel. Increase the signal to ensure that the PFPD's signal is not limited by the input noise of the Chromatographic Data System (CDS), so that accurate quantitative measurements result (i.e. the noise of the PFPD signal is not buried under the CDS noise). Increasing the multiplier decreases the upper bound proportionally, and peaks will be truncated (flat topped) if they exceed the upper limits of the DAC. (See Chapter 15, Noise Sources and Output Signal Optimization, for a more detailed discussion of noise.) (See Appendix B for more details on setting the offset properly.)
Output Zero	Shows the instantaneous average baseline between the gates set for the reported output channel.

Output Set	Sets the zero offset for the output channel, which can be either the output zero or some lower value specified by the operator. Specify the lowest obtainable output zero in the tick-tock mode as the set value to maximize the dynamic range for chemiluminescent signals.

The function of each button along the bottom of the Board/Channel Parameters screen follows:

Ok	Transfers all board and channel parameters to the detector controller and exits the screen.
Cancel	Aborts the current entry including any changes made by clicking **Apply** and replaces them with the last saved entries. Click **Cancel** to exit the screen without applying any changes.
Apply	Transfers all board and channel parameters to the detector controller. Click **OK** to save these changes and exit the screen.

NOTE: Changes are automatically transferred to the 5383 unit and saved to the EEPROM when **OK** or **Apply** is pressed.

Timed Events Screen

To access the Timed Events screen, click the **Timed Events** icon on the toolbar (Figure 4.4).

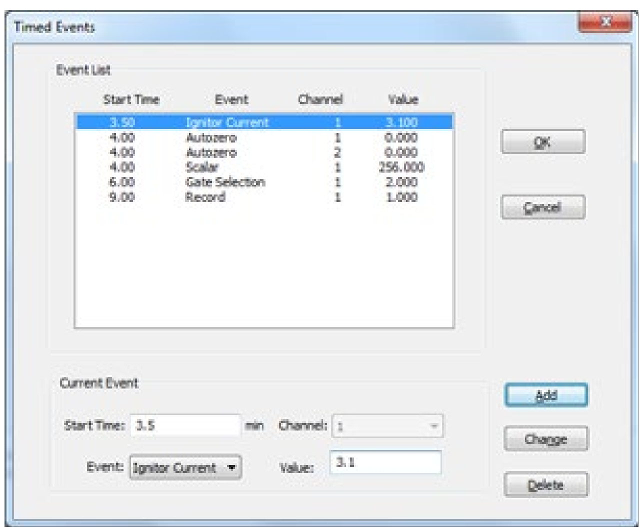


Figure 4.4. Timed Events screen

The detector controller's remote start initiates timed events, which is activated by a contact closure supplied either by the GC or an external remote start switch. The event list shows the current timed events. Modify the value of any current timed event parameter by clicking the row in which the parameter to be modified occurs and then change the value of the desired parameter in the appropriate field shown in the lower Current Event box. The function of each field in this box follows:

Start Time	Sets the time in minutes from the start of the analytical run for starting the associated event. The time must be greater than or equal to 0.01 minute.
Event	Sets a timed event from a list by clicking the **dropdown arrow** next to the Event field. Scroll down through the list until the desired event is located and then click it. Figure 4.4 shows an example of each event that can be selected. The selected events initiate at the specified times.
Channel	Specifies the channel on which the event occurs.
Value	Sets the value that applies to the selected event. The unit of measure for the value varies by event. For a recording event, the value is the start recording or stop recording commands.

Event	Description	Value
Auto-Zero	Sets the zero offset on the selected channel.	On/Off
Scalar	Changes the multiplier of the output on the selected channel by the specified value. **NOTE:** Changing the multiplier will alter the signal-to-noise ratio. (See Chapter 15, Noise Sources and Output Signal Optimization, for a more detailed discussion).	Select values of 1, 2, 4, 8, …, 4096.
Gate Selection	Specifies which gate mode, listed in the gate parameters list, links to the selected channel mode (see below).	See the list of gates defined on the Gate Parameters Screen.
Record	Records the raw chemiluminescent data between the specified start and stop times (in minutes) into a data file.	On/Off
Ignitor Current	Sets the current delivered to the PFPD ignitor.	0...3.0A
Reset to Ready	Resets the 5383 to ready condition and stops processing the current run.	n/a

The function of each button along the right side of the Timed Events screen is described below:

OK	Transmits the event list to the detector controller and exits the Timed Events screen.
Cancel	Disables all changes made to the timed events parameters and exits the screen without downloading the changes to the detector controller.

Add	Appends a timed event to the event list.
Change	Modifies current timed event without adding a new mode to the event list.
Delete	Deletes the selected timed event.

NOTE: Changes are automatically transferred to the 5383 unit saved to the EEPROM when **OK** is pressed.

Chapter 5 PulseView Analyzer Software

The PulseView Analyzer software package allows the operator to perform post-processing analysis of data from the 5383 PFPD. This program is used to reprocess portions of, or all, the run data using different PFPD operational parameters. Data reflecting the changes does not require rerunning the chromatographic analysis. This allows the optimal parameters to be adjusted to find the ideal parameters for selectivity. Post-run analysis facilitates method development, dual gate optimization, and support for structural information of unknown compounds.

PulseView Analyzer provides the following features:

- Provides post-run display of emission scans, gate parameters, and chromatograms.
- Allows the operator to review time emission profiles to obtain some heteroatom structural information of unknown compounds.
- Allows the operator to create or modify gates to optimize sensitivity and selectivity.
- Permits off-line optimization of the dual gate modes to enhance inter-heteroatom selectivity.
- Permits reanalysis off-line of chromatographic runs using different PFPD parameters.

PulseView Analyzer Main Screen

The PulseView Analyzer main screen is neatly organized for efficiency. It consists of a button toolbar as well as separate graphics displays for the time emission profile pulse and the chromatogram. See Figure 5.1.

- The top window is the selected time emission profile for the pulse indicated by the cursor line in the chromatogram (displayed on the PulseView Analyzer Screen).
- The lower window is the resulting chromatogram for the entire analytical run, calculated from the specified gate, board, and channel parameters from all of the collected time emission profiles.

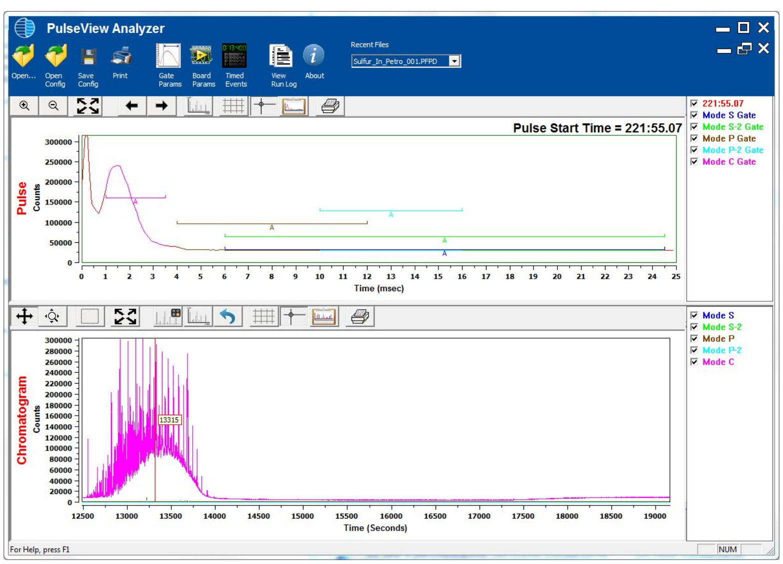


Figure 5.1. PulseView Analyzer Main Screen

The analysis time is displayed on the first line of the legend. The vertical axis (Y) on the chromatogram represents the (peak) intensity, and the horizontal axis (X) is the retention time of the peak in seconds. Exact retention time information is not available unless the 5383 Detector Controller is synchronized (via remote start) with the GC's run start.

The time emission profile (pulse) for a specific point on the chromatogram are viewed three ways: (1) clicking the selected area/time of the chromatogram, (2) clicking the appropriate arrow button ( or ) on the toolbar, or (3) pressing the arrow keys on the keyboard until the vertical cursor line is on the specific point.

The cursor line allows pulse emission viewing for each line of the chromatogram. Holding the right-arrow or left-arrow key down moves the cursor line across the chromatogram. By viewing each pulse, elements and their ratios are identified. For more information, see Analyzing and Viewing Data.

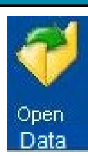
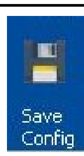
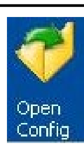
The horizontal colored lines in the time emission display indicate the gate settings for the corresponding mode. To add or remove these indicators, simply click the appropriate check box in the legend.

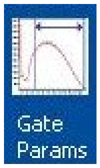
Note: Chromatograms for up to five gate settings are displayed simultaneously. Each chromatogram is displayed or hidden by clicking the mode of interest in the chromatogram legend.

Button Bar Features

All of PulseView Analyzer's functions are accessed by via the toolbar. Table 5.1 shows the complete toolbar feature list.

Table 5.1. Summary of the PulseView Analyzer Button Bar

Button	Purpose
	Opens an existing .PFPD data file (created in PulseView Monitor) for graphing/viewing.
	Allows the operator to save the current (as edited) configuration parameters by specifying a file name and directory location to save the current configuration settings. These can be recalled and applied later in PulseView Monitor using the Open Config button.
	Allows the operator to select which saved configuration file (.cnfg) to open and apply to the current data set. The changes will be populated in the various screens and will also cause the chromatogram to be recalculated and updated.

	Allows the operator to print all of the configuration parameters for the Gates, Board/Channel, and Timed Events screens for record-keeping purposes.
	Accesses the Gate Parameters screen, which is used to set gate values and identify gate mode labels. (For more information, see Gate Parameters Screen section in Chapter 4.) **NOTE:** All values changed can have an effect on the chromatogram.
	Accesses the Board/Channel Parameters screen, which is used to set the parameters for the PFPD Board and the Channel 1 and Channel 2 output signals to the GC. (For more information, see Board/Channel Parameters Screen section in chapter 4.) **NOTE:** Only the channel values changed can have an effect on the chromatogram.
	Accesses the Timed Events screen, which allows the operator to program events to occur based on the time since the start of the run. (For more information, see Timed Events Screen section in chapter 4.) **NOTE:** No changes will have an effect on the chromatogram.
	Displays the Run Log screen, which reflects all changes made to the PFPD settings with a time and date stamp.
	The About screen provides system software and firmware version information. It also provides real-time feedback of a few status variables.

Loading a Data File

Data files used in PulseView Analyzer are created in the PulseView Monitor program supplied with the 5383 PFPD. These files are created either manually (from the main screen, by pushing the **Record** button) or automatically (by using the timed event function). Once collected in PulseView Monitor, the *.PFPD files is opened and used with PulseView Analyzer.

1. Click the **Open Data** button in the toolbar.

2. Select the data file that you want to open, and click **OK**. This will load the selected data file to PulseView Analyzer, including the Configuration parameters used in the PulseView Monitor program when the .PFPD file was created.

Note: PulseView Analyzer calculates a chromatogram from the stored pulses (time emissions) in the data file for every gate mode in the current gate table (i.e., for each mode that was in the Gate Parameter List in PulseView Monitor when the file was created) (see Gate Parameters Screen in Chapter 4 of this manual). The progress of chromatogram generation is indicated in the lower-left corner of the main screen.

3. After a data file is loaded, a new configuration file may loaded and applied, if needed. To do so, click the **Open Config** button in the toolbar.

Note: The chromatograms will be recalculated whenever a new configuration file is opened.

Note: If the data was collected using the PulseView Monitor Record button and not the GC remote start, the time scale used for the chromatogram in PulseView Analyzer does not match the one generated by the GC data handling system.

Analyzing and Viewing Data

Use the cursor line in the chromatogram display to select each pulse emission in the chromatogram to view them individually (see Figure 5.1).

By observing individual pulses, the operator obtains several types of useful information. The time emission profile provides the flame characteristics in the PFPD. The emission is observed from the detector to optimize the flows and tuning. Cleanliness of the detector is seen by noting the time emission profile in the early (1.5–3.0 msec) time frame.

Additionally, observe the pulses to see elemental information for peaks of interest. To determine whether a peak has S or P in it, look at the pulse time window. Relative concentrations of each element in a specific peak can also be determined by the emission time profile, as shown in Figure 5.2. This is helpful when trying to determine unknown composition.

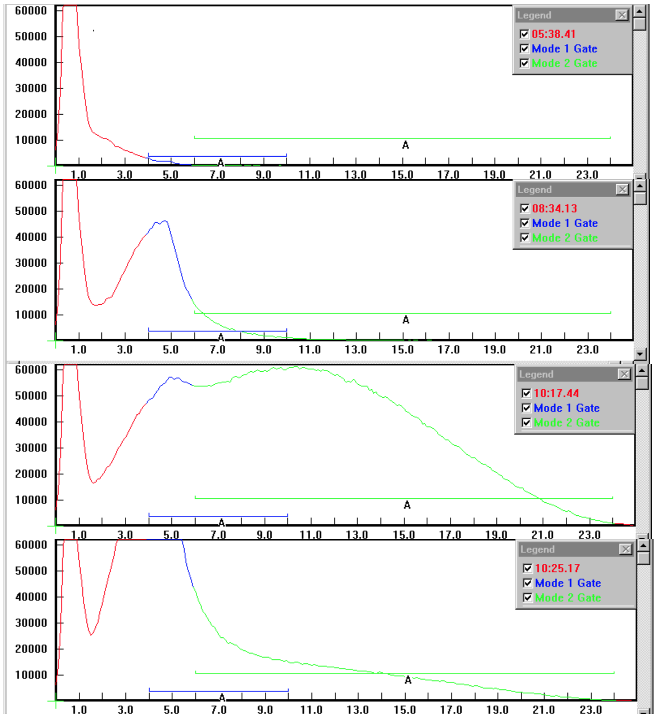


Figure 5.2 Using PulseView Analyzer to Obtain Heteroatom Composition Information

1. Place the cursor on the peak then click the specific peak on the chromatogram or by using the arrow keys on the keyboard to move the cursor line to the specific peak.

Note: To zoom in on a specific area on either the emission profile or the chromatogram, hold down the left mouse button and drag the cursor over the area to be zoomed in on. Release the mouse button. To return to the original view, go to the View menu and select **Restore**.

2. If necessary, isolate the specific gate by checking/unchecking the mode (gate) in the legend.

Adjusting Analysis Parameters

PulseView Analyzer is used to change the gate parameters and the resulting chromatogram will be displayed. Once optimized, a new configuration file can be generated and used in PulseView Analyzer. The results of the new parameters are viewed and adjusted without the need to rerun the analysis. This chromatograph is reanalyzed off-line using only a single chromatographic run.

1. Select the appropriate screen to be accessed (Gate Mode, Timed Events, or Board/Channel).

2. Make any necessary changes.

Note: All fields cannot be modified. For information on the Gate Parameters Screen, Timed Events Screen, and Board Channel Parameters Screen, see Chapter 4.

For example, to change a gate parameter, go to the toolbar and click the **Gate Mode** button to access the Gate Parameters screen. Click the **mode to be modified**, and make the changes in the specific parameter(s) to be changed.

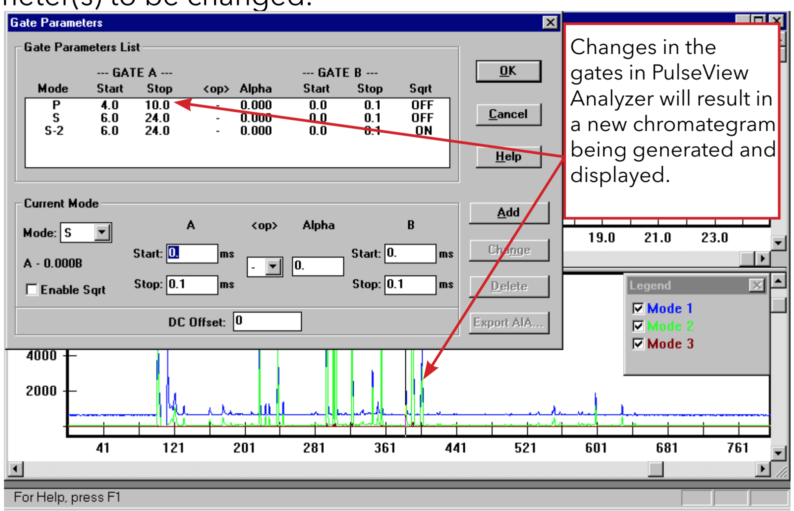


Figure 5.3. Post-Run Reprocessing of Gate and Timed Events Parameters

3. Click **Change** for the changes to take effect in the gate table.

4. Click **OK**. PulseView Analyzer will recalculate the chromatogram using the new parameters.

Creating New Configuration Files

To save the modified parameters in a new configuration file for use in PulseView Monitor, click the **Save Config** button in the toolbar. Enter the name and path of the configuration file to be saved, then click **Save**.

This configuration file can then be opened and used in PulseView Monitor. Alternatively, the new PFPD parameters are simply manually entered directly into the PulseView Monitor program.

Optimizing Using Dual Gates

Dual Gate Mode of operation eliminates interferences between two species. This technique improves the selectivity of the PFPD significantly. The data is enhanced using the dual gate subtraction mode. For more information, see Appendix A.

1. Load a data file (see Loading a Data File in this chapter).

2. From the toolbar, select **Gate Mode** button, Then, select the **mode to optimize**. Change the **Alpha value** and the **Gate B parameters** as needed. Click **Change**.

3. Click **OK**. The chromatogram will be recalculated and displayed using the new gate parameters. (See Figure 5.4.)

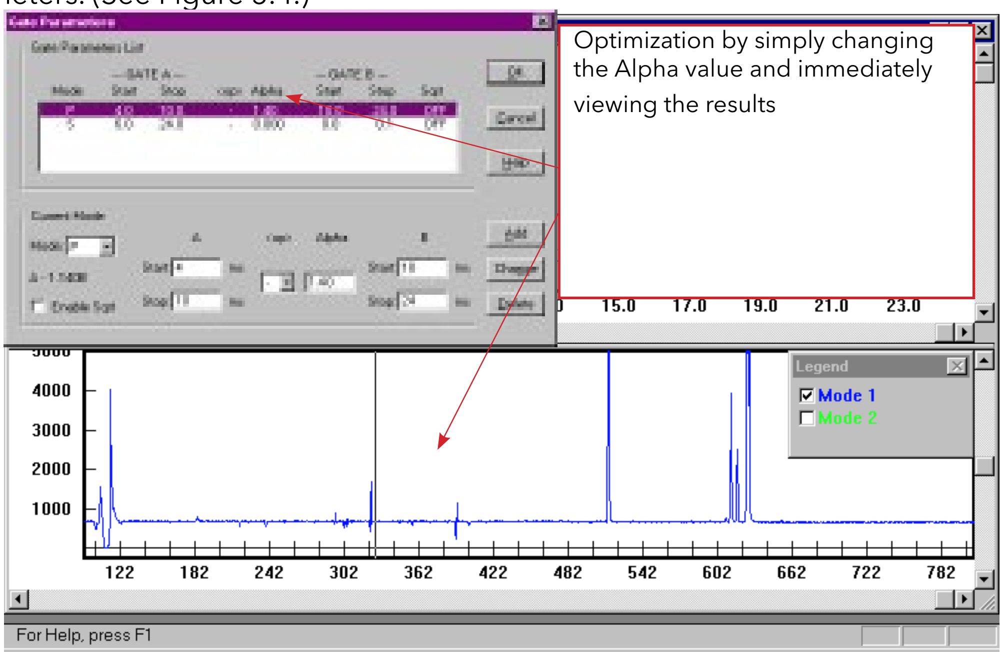


Figure 5.4. Post-Run Optimization of Gate Alpha Values

4. Evaluate the chromatogram. Repeat steps 2–3 until the proper Alpha value is obtained. (See Figure 5.5.)

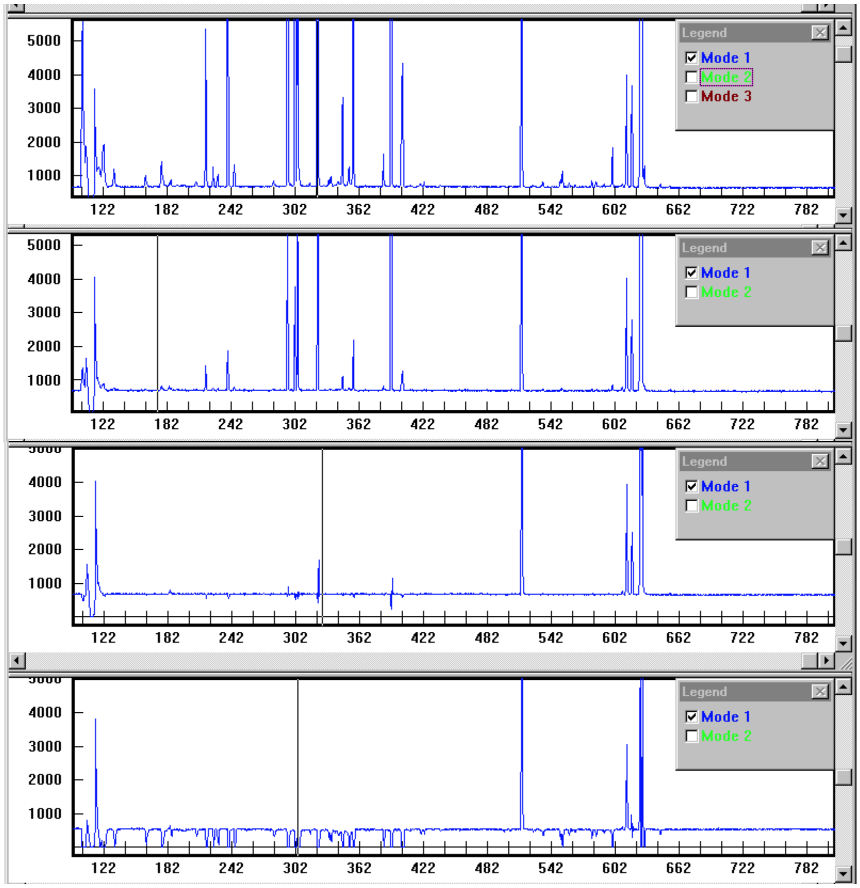


Figure 5.5. Post-Run Optimization of Dual Gate to Eliminate S Interferences in a P-Optimized Analysis

5. Once optimized, click **Save Config** on the toolbar to save the new value(s). Enter the name of the configuration file to be saved, then click **Save**. The new configuration file can now be called up in PulseView Monitor and used for future runs. Alternatively, manually enter the final optimized parameters into the PulseView Monitor Gate Parameter Screen.

Exporting the Chromatogram

Each chromatogram displayed in the PulseView Analyzer screen can be exported as a new GC data file in a standardized AIA format. These files are imported into most chromatographic data systems that accept AIA file formats. This permits the operator to view, reprocess, print, or reintegrate the GC run under the new PFPD parameters without rerunning the sample.

Note: Caution must be taken when reanalyzing post-processed runs as calibration procedures must take into account any PFPD parameters that have been changed.

1. From the toolbar, click **Gate Mode**. This will access the Gate Parameter screen.

2. In the Gate Parameters List, select the desired gate mode to be exported as an AIA file (see Figure 5.6). One of the modes in the Gate Parameters List must be selected to activate the Export AIA button.

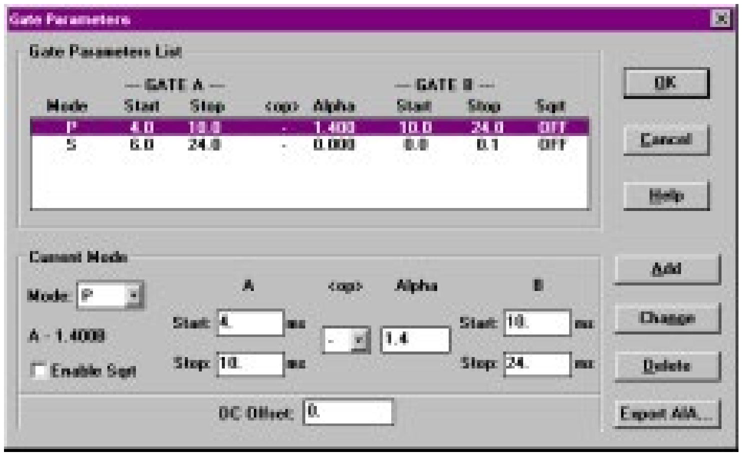


Figure 5.6. Gate Parameters Screen

NOTE: The DC Offset, can only be changed for the modes specified in the Board Channel screen. See details for changing that Zero value offset the Board Channel Screen section in Chapter 4. All other channels will have an assumed DC Offset adjustment of 0.0.

3. Click **Export AIA**. This will access the Export AIA Data screen (Figure 5.7).

4. Enter in all the necessary information. Fields that may be modified are displayed with a white background. After all the necessary information has been entered, click **Save As**.

5. Enter the name of the export file to be saved, then click **OK**.

6. To import the file into a data system, refer to the data system's operator's manual.

7. Reprocess the chromatogram as desired using the GC data system.

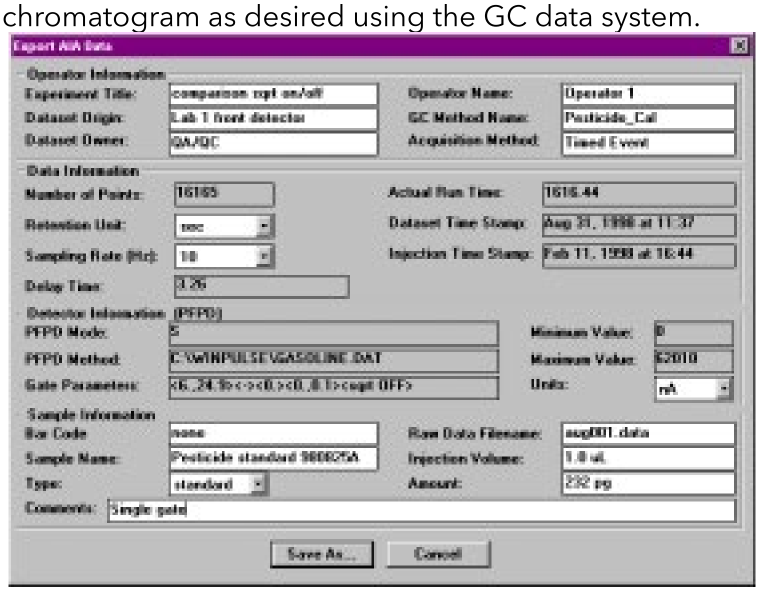


Figure 5.7. Export AIA Data Screen

Troubleshooting Time Scale Issues

If data is collected using the PulseView Monitor Record button, the time scale used for the chromatogram in PulseView Analyzer will not match the one generated by the GC data handling system. Three different types of data file issues can result.

- **PulseView Monitor is in Run and the Clock is Updating**
  The time base may not correspond to the chromatographic time being recorded. Relative time differences between PFPD pulses will be accurate; however, the absolute time will appear to be incorrect.

- **PulseView Monitor Record is Pressed Prior to GC Start (with start cable connected)**
  When record started prior to run start, an injection will be made into the GC and the detector controller will detect a run start signal. PulseView Monitor closes the file that is currently open, increments the counter, and opens a new file to start recording. The first time emission profile will occur immediately after the run start signal is detected by the detector controller.

- **PulseView Monitor is in Ready and Does Not Advance to Run**
  The elapsed time from run start remains at zero (0) for every time emission data set. When PulseView Analyzer opens this file, the secondary time base (time elapsed from the previous pulse) will be summed to generate the time base.

Additional PulseView Analyzer Uses

For additional uses for the PulseView Analyzer, see Appendix A.

Chapter 6 Operation

This chapter describes operating the 5383 PFPD for GC analyses.

Starting the PFPD System

CAUTION: An excessively high ignitor current shortens the ignitor lifetime.

WARNING: Use proper eye protection to avoid eye injury.

Once the PFPD assembly, detector controller, pneumatics, and PulseView Monitor are installed, and all of the necessary electronic connections have been made, the system is ready to start. Before starting the system, ensure the GC is correctly configured for the application. The PFPD is designed to use low column flow rates. Obtain maximum sensitivity with flow rates of 1–2 mL/minute. The maximum flow rate is 5 mL/minute, but there may be some sensitivity loss. Flow exceeding 5 mL/minute may be possible only when H_2 is used as a carrier gas. When the carrier gas flow is 4–6 mL/minute, use the 3-mm combustor.

Follow these steps for the initial PFPD setup:

1. Plug in the GC's power cord and turn on the GC.

2. If the initial gas flows were not set during installation or if they were adjusted after installation, follow the sequence below, or refer to the PFPD Quick Reference Guide:

   - Verify the air and H_2 supply lines are pressurized to at least 60 psi. Open the valves on the cylinders supplying H_2 and air to the PFPD's pneumatic system.
   - Replace the flame arrestor located on top of the detector cap with the barbed fitting (PN 202077) and attach a flow meter to the fitting.
   - Refer to Table 4.3 Flow Controller Settings.
   - Replace the barbed fitting with the flame arrestor.

3. Using the GC's electronic keyboard, set the detector to the appropriate temperature and allow 15 minutes for the detector to reach a steady state. To detect sulfur, set the PFPD temperature to 250 °C. To analyze sulfur pesticides, set the PFPD temperature to 300 °C.

4. Once the detector base temperature reaches 100 °C, turn on the power to the PFPD using the switch on the back panel of the detector controller. The green ON LED on the front panel of the detector controller lights up when the power is in the ON position.

5. The PFPD should begin pulsing within a few seconds (the time required for purging the associated gas lines). An initial sharp pop may be heard due to the flame propagating out through the vent in the detector cap, especially if the gas flows are turned on before the power to the PFPD is turned on. The pulses should settle down to a steady rate.

6. If PulseView Monitor is not already running, activate the PulseView Monitor software program on the attached PC by double-clicking the **PulseView Monitor** icon.

7. Click the **Board Params** button to access the **Board/Channel Parameters** screen. Verify the ignitor current is set to 2.8 A.

8. If the PFPD does not begin pulsing or pulses erratically:

   - Using proper eye protection or a mirror, verify the ignitor coil is glowing orange by looking down through the flame arrestor in the ignitor cap. If it is not glowing, increase the ignitor current until the ignitor coil is pale red.
   - Turn the power switch off on the front of the detector controller.
   - Check gas connections for leaks and verify initial gas settings.
   - Restart the ignitor by turning he power supply on to the PFPD. (If PulseView Monitor is activated and the power supply is still on, the ignitor can also be restarted by setting the **Ignitor Current** to 3.3 A in the Board/Channel Parameters screen.) Reset the ignitor current to 2.8 A once a regular pulse is obtained.

9. If the PFPD still does not pulse or continues to pulse erratically, confirm that the gas flows are correct. If the settings of the column carrier gas or the H_2 gas flow are set too high relative to the air flow, ignition may be difficult due to excessive cooling of the ignitor coils. Reduce the column gas or H_2 flow rates, or alternatively, increase the Air 2 gas flow slightly.

10. Verify the PMT voltage is set to the appropriate level.

    - Go to the Board/Channel Parameters screen and set the **PMT Voltage** to 600.
    - While monitoring the real-time emission display in PulseView Monitor, adjust the PMT voltage so that the first sharp emission, at ~0.5 msec, is just below 1,600,000. The PMT voltage will usually fall between 500–650 volts.
    - Set the Ignitor current to 2.8.
    - Set the Trigger Level to 200.
    - Set the Interpolation Level to 800,000.
    - Save the board parameters.

Optimizing the Combustor Flow (Sulfur)

Manipulating the flow rate and mixture of combustor gases (H_2 and Air 1) maximizes the chemiluminescent emission lifetimes of the specific elements of interest. The chemiluminescent emission lifetime of each element starts after a particular delay following the passage of the flame through the combustor. The emissions of some elements are delayed more than others. For example, sulfur emissions are delayed more than phosphorus and have a longer lifetime. The following focuses on maximizing the lifetime of phosphorus and sulfur chemiluminescent emissions.

Figure 6.1 illustrates background chemiluminescent emission when no analyte is injected into the GC column. Two major emissions are typically seen, but several smaller emissions may also be observed. The first main emission at ~0.5 msec is due to flame propagation through the detector cap and hitting the combustor top. The second main emission at 1–3 msec is due to the OH*, C2*, and CH* emissions and can be used to evaluate specific aspects of detector performance. A spike to the right of the second main emission, at ~4 msec may occur when the flame causes the top of the GC column to fluorescence.

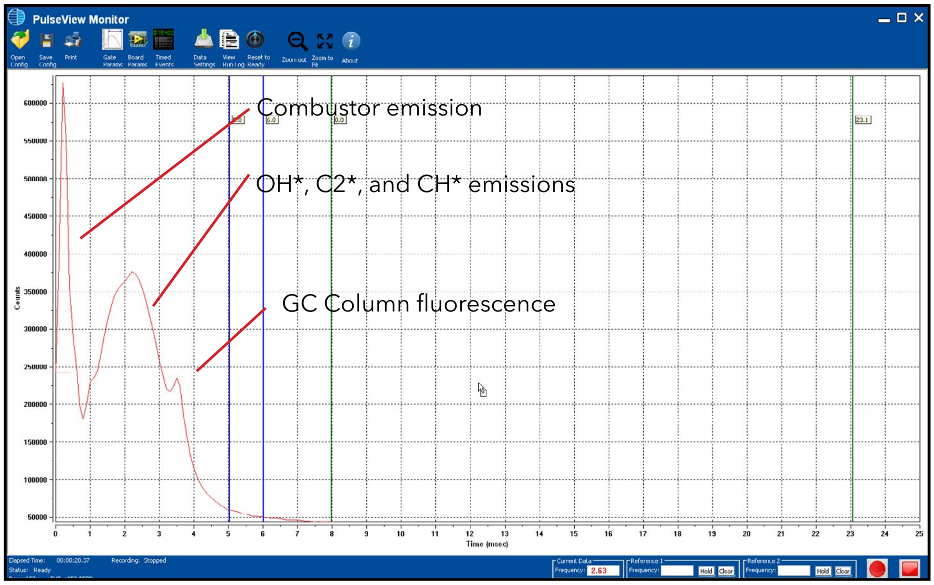


Figure 6.1. Primary Peaks and Secondary Spikes of PFPD Fluorescence

Optimize the combustor and wall gas flow rates for the installed column, column gas flow rate, and detector temperature by delivering analyte to the PFPD. Easily optimize either by temporarily installing a standard permeation source or by making multiple manual injections of a standard analyte.

Using a Permeation Source

OI Analytical offers permeation kits for sulfur (PN 290411) and phosphorus (PN 290429) that include a permeation source and associated hardware.

Confirm that the blue, BG-12 filter is installed and introduce a constant source of sulfur for turning. For example, install a permeation source between the injector and the GC column to deliver a steady analyte concentration to the combustor. Because the permeation rate is a function of temperature and may be too low at room temperature, adjust the GC oven temperature to obtain a readily-detectable emission in PulseView Monitor. However, excessive heating of the permeation source may damage the permeation device. Do not exceed the upper temperature limit recommended for the permeation source being used. The recommended temperature range for the permeation sources supplied by OI Analytical (PN 290452 for sulfur source, PN 290460 for phosphorus source) is 35–60 °C.

NOTE: When using a permeation source for the first time it may be necessary to heat it at 60 °C for several hours to obtain detectable emissions.

NOTE: OI Analytical recommends tuning the gas flows with sulfur regardless of the application.

Optimize the PFPD response once a steady permeation rate is achieved. To optimize PFPD operation, set the column flow to the minimum flow rate that occurs during the chromatographic analysis. Failure to do this may result in the PFPD entering tick-tock mode as column flow decreases due to increasing GC oven temperature, elevating PFPD noise. For this reason, it is important to maintain constant column gas flow with an EPC gas flow regulator. Install the permeation device and optimize the PFPD response using the following procedure.

1. Remove one end of the previously installed GC column from the detector. (If installing a sulfur permeation tube, the injector side may be used, but phosphorus tubes must be installed on the detector side of the column.)

2. Install one end of an additional short column piece (about 18" long) into one of the two inlets of the permeation chamber using a reverse ferrule nut (PN 223057) and an appropriate ferrule (PN 208330 for a 0.25-mm I.D. column, PN 196113 for a 0.32 mm I.D. column).

3. Install the other end of the short column piece either into the detector or the injector. Remove the detector cap, combustor tube, and detector body (see Chapter 6 Maximizing Sulfur Response). Ensure the end of the short column piece inserted into the detector projects 0.5 mm to 1.5 mm above the top of the combustor support (Figure 3.38). Rebuild the detector assembly (see Chapter 4 Installing the Combustor and Reassembling the PFPD).

4. Install the end of the GC column into the other inlet of the permeation chamber using a reverse ferrule nut and ferrule.

5. Slowly increase the oven temperature until sufficient chemiluminescent signal is observed in PulseView Monitor. Do not set the temperature so high that the signal amplitude begins to approach the maximum signal handling capacity of the PFPD (i.e., the signal should be well below 2,000,000 counts in PulseView Monitor).

6. If a chemiluminescent signal is not obtained within the permeation source's recommended temperature range, check for gas leaks at the column connections to the permeation source, injector and detector, and along the PFPD assembly. If the short column section is attached to the detector, ensure the column does not extend too far into the detector body (i.e., more than 1.5 mm above the top of the combustor support).

7. After a chemiluminescent signal from the diffusion analyte becomes detectable in PulseView Monitor, allow 10–15 minutes for the oven temperature and permeation source to stabilize before optimizing the PFPD's response. If the PFPD or GC oven temperatures are changed, let the permeation source stabilize for 10–15 minutes after reaching the new temperature.

8. After the GC oven temperature stabilizes, check the pulse frequency box on the PulseView Monitor Main screen to ensure the PFPD is pulsing at about three to four times per second. When the pulse rate begins to exceed about 4.5 Hz, the display on the PulseView Monitor screen turns red indicating that the data generating frequency exceeds the PFPD's data handling capacity. Change the pulse by adjusting the Air 2 flow rate.

CAUTION: Never heat the permeation source to more than 60 °C.

9. The H_2:Air 1 flow ratio should have been achieved by the initial H_2 and Air 1 flow settings made prior to starting the PFPD. If this ratio is correct and a sulfur permeation source is connected, a chemiluminescent signal similar to that shown in Figure 6.3 should be observed. In particular, note the start and end of the OH*, C2*, and CH* emission and the start and end of the sulfur emission. (If using a phosphorus permeation source, the start and end of the chemiluminescent emission occurs considerably sooner than shown in Figure 6.3.)

10. If the H_2:Air 1 ratio is either significantly too high or too low, the sulfur emission lifetime rapidly decreases, becoming shorter in time and will not extend all the way to 24 msec. Hold the Air 1 flow rate constant and adjust the H_2 flow rate to maximize the lifetime of the sulfur emission profile. Adjust the gas flows to maximize the delayed sulfur emission. Note that as the H_2:Air 1 ratio is increased, the amplitude of the OH*, C2*, and CH* emission (second emission from the left) decreases. Err on the Air 1-rich side of the optimum than on the H_2-rich side for better stability and reduced quenching.

11. For a 2-mm combustor, the sulfur emission should extend all the way to 24 msec (as shown in Figure 6.2) For a 3-mm combustor the emission may be shorter, extending only to 20 msec. For either combustor, H_2:Air 1 ration should be adjusted so that the sulfur emission extends as far as possible.

12. Check that the detector pulse rate is still in the 3–4 Hz range. Adjust Air 2 if it is outside of this range.

13. When the optimal gas setting is attained, verify the PFPD is close to tick-tock mode by closing the fine adjust needle valve (clockwise) until the PFPD just goes into tick-tock mode (Figure 6.2). In tick-tock mode, only every second flame propagates through the combustor. In PulseView Monitor, observe a sulfur emission only every second pulse. Chromatograms obtained during tick-tock mode contain significantly higher noise. Therefore, once tick-tock mode is obtained, open the fine-adjust needle valve until tick-tock stops. Open an additional 1/2–1 full turn.

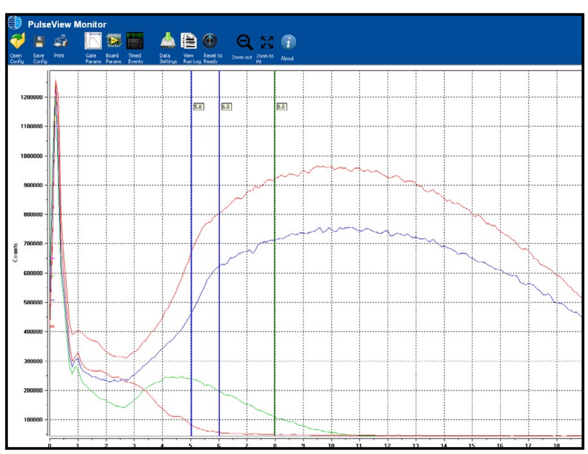


Figure 6.2. Tick-Tock Mode and Stable Florescence

14. Disconnect the permeation source from the injector or detector. Reconnect the GC column to the injector or detector.

NOTE: Optimize for phosphorus compounds using the sulfur optimization process described above. Then add an additional 1 mL/minute to Air 1 and install the yellow phosphorus filter.

15. Change the pulse rate over a narrow range by adjusting the Air 2 flow rate. For a significant change of the PFPD pulse rate, proportionally increase or decrease all the gas flow rates as the pulse rate linearly depends on the total gas flow.

Using Multiple Manual Injections

If a permeation source is not available, optimize the PFPD's detectivity by making multiple isothermal injections of a test analyte.

1. Inject a sample containing the analyte of choice into the GC column. With most solvents, the flame may not propagate when the solvent elutes from the column due to excessively rich fuel conditions. As the solvent concentration decreases in the PFPD to a sufficiently low level, the flame reinitiates.

2. Monitor the height of the analyte's chromatographic peaks on the data integration device (e.g., software such as ChemStation™).

3. With each subsequent injection, increase the H_2 flow, monitoring both the background emission and the sulfur emission. The emission lifetime will increase to a maximum of 24 msec then start decreasing. The H_2:Air 1 in PulseView Monitor gas mixture is optimized when the sulfur emission extends to 24 msec. Reduce quenching by setting a slightly air-rich mixture.

4. Use PulseView Monitor to archive data for comparison purposes when making multiple manual injections. Use the optional PulseView Analyzer software to observe the stored data.

5. After the optimum H_2 flow is established and set, check that the detector pulse rate is still in the 3–4 Hz range.

6. When the optimal gas setting is attained, verify the PFPD is close to tick-tock mode by closing the fine adjust needle valve (clockwise) until the PFPD just goes into tick-tock mode (Figure 6.2). Open the fine adjust needle valve until tick-tock stops, then open an additional 1/2–1 full turn.

NOTE: To reduce the time required to optimize the combustor detector flow using the multiple injection method, make injections in rapid succession. To use this technique successfully, initially determine the difference in the elution times of the solvent and analyte of choice. The subsequent multiple injections must be completed within that time period or spaced in time so that the analyte does not elute simultaneously with the solvent from a subsequent injection.

7. Optimize the PMT voltage and trigger level. See step 14 of "Starting the PFPD System" in this chapter, or as described in the PFPD Quick Reference Guide.

Notes on Optimizing Combustor Gas Flows

The following notes are offered to assist with conditions that may be observed while optimizing the combustor gas flows. For additional assistance, contact OI Technical Support Department at (800) 336-1911 or (979) 690-1711.

Balancing the WALL and COMB Gases

Observe the chemiluminescent emission signal displayed in the PulseView Monitor Main screen. If only one background emission is observed when no sample has been injected, the flame may not be propagating fully through the combustor. Turn the fine adjust needle valve counterclockwise to increase the H_2:Air 1 mixture flowing through the combustor. Also, reduce the Air 2 flow rate to decrease the flow rate of the wall gas mixture relative to the combustor gas mixture.

Adjusting the Fine Adjust Needle Valve

Slowly turn the fine adjust needle valve clockwise to reduce the flow of the H_2:Air flow into the combustor until the PFPD goes into tick-tock mode. (See Chapter 1, Operating Principles for more information.) When the PFPD goes into tick-tock mode, turn the fine adjust needle valve counterclockwise until the PFPD goes out of tick-tock mode. Then turn the valve one-half turn counterclockwise for stable PFPD operation.

Effect of H_2 Flow

Increasing the H_2 flow gradually cools the gas mixture in the combustor until the flame no longer fully propagates into the combustor. This manifests as an increase in the time spread and a decrease in the amplitude of the flame background OH*, C2*, and CH* emission until the second emission is no longer visible. Simultaneously, the sulfur emission rapidly decreases in lifetime and emission intensity (Figure 6.3). Slowly decreasing the H_2 flow results in the sulfur emission building back up to a maximum of emission amplitude and lifetime. Observe a similar dynamic with a phosphorus emission, but the optimal H_2:Air 1 ratio mixture for phosphorus is lower (0.9–1.0) than that for sulfur (1.1–1.2).

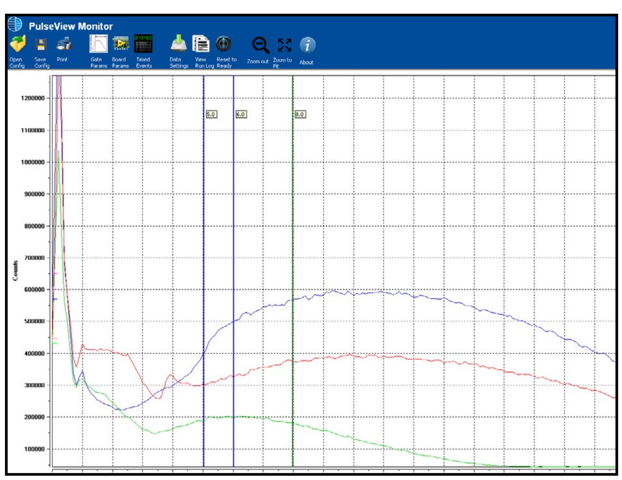


Figure 6.3. Effect of H_2:Air Ratio on Chemiluminescent Intensity and Sulfur Emission Lifetime

Effect of Column Flow

Increasing the column flow rate beyond the optimal rate has a similar but smaller effect on the chemiluminescent amplitude and lifetime resulting from analyte combustion, because increasing the helium or nitrogen carrier gas flow also cools the propagating flame. However, the carrier gas flow rate is about an order of magnitude less than that of H_2, and the thermal conductivity of helium or nitrogen is less than that of H_2.

Optimizing PFPD Detectivity and Selectivity

In addition to the combustor gas composition, several other PFPD parameters can be used to optimize PFPD response. These include optical filters, PMTs, gate settings, combustor diameter, and detector temperature. Because the two output signal channels operate independently, specify different start and stop times for their gates, optimizing the detector for more than one condition. For example, set the gate start and stop times to maximize detectivity by including most of the analyte's chemiluminescent emission lifetime within the gate. (See Chapter 13, Defining Detectivity for calculating detectivity and sensitivity of sulfur and phosphorus modes.)

Alternately, set gate times to maximize selectivity by limiting the gate width to a segment of the analyte's chemiluminescent emission lifetime that is independent of interference from other chemiluminescent sources. Setting gates for two elements (e.g., phosphorus and sulfur) with no gate overlap may not be possible because detectivity may be excessively compromised for one or both elements. When gate overlap is inevitable, use dual gate techniques to increase selectivity. (See Chapter 14 Dual Gate Capability and Alpha Calculation for a detailed discussion of the dual gate technique.) Obtain maximum sensitivity and selectivity with the appropriate filters and PMTs to match a given application. For assistance in properly configuring the PFPD to a specific application, call OI Analytical's Technical Support Department at (800) 336-1911 or (979) 690-1711.

Optimizing for Detectivity

Maximizing Phosphorus Response

Maximize the PFPD's detectivity for phosphorus using the following steps:

1. Install a 3-mm combustor. (See Installing the Combustor and Reassembling the PFPD.)

2. Because HPO* is the species emitting the chemiluminescence, reduce the H_2:Air 1 ratio of the combustor gas mixture relative to the optimal sulfur setting by increasing the Air 1 flow rate by ~ 1 mL/minute.

3. Increase the detector temperature to 300 °C. The uniformity of the phosphorus response decreases below this temperature while phosphorus detectivity may decrease up to about 325 °C. Therefore, do not set the detector temperature any higher than that required to preserve the chemiluminescent peak shape and lower than that needed to maintain column integrity.

4. Adjust the fine adjust needle valve so that it is half a turn counterclockwise from the point at which the PFPD goes out of tick-tock.

5. In the Gate Parameters screen, set the integration gate's start and stop times (OI Analytical recommends 4–5 ms and 15 ms, respectively) to optimize phosphorus.

6. Optimize the PMT voltage and trigger level. See step 14 of "Starting the PFPD System" in this chapter.

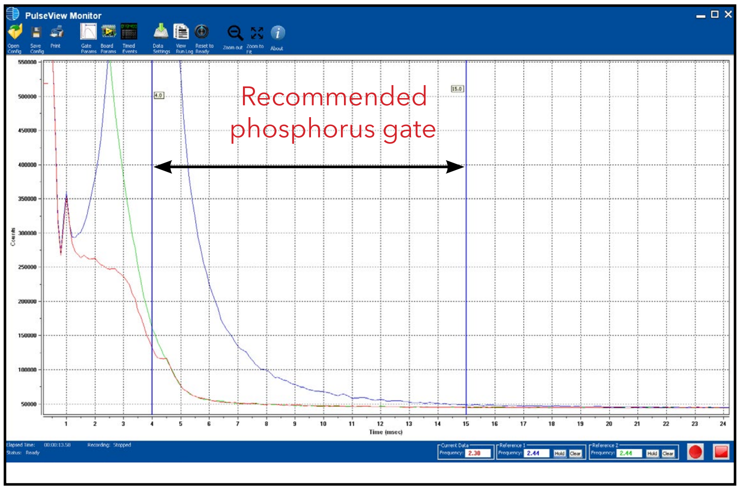


Figure 6.4. Phosphorus Emission and Gate Settings for Maximizing Phosphorus (The PMT Voltage Was Set to 400 V)

Maximizing Sulfur Response

Sulfur exhibits a quadratic chemiluminescent response (i.e., the response is a function of the square of the sulfur concentration). For detecting trace levels, increase sensitivity by maximizing sulfur concentration within the combustor just as the flame begins to propagate through it. Conversely, for detecting high concentrations, decrease the overall sulfur concentration in the combustor during flame propagation.

For trace-level analysis, increase sulfur concentration within the combustor and the PFPD's detectivity for sulfur using the following steps:

1. Install the 2-mm combustor. (See Installing the Combustor and Reassembling the PFPD.)

2. Set the detector temperature to 250 °C. Using higher temperatures for some applications if needed may result in a small sensitivity decrease.

3. Set the combustor gas composition to the optimum H_2-rich mixture.

4. Set the fine adjust needle valve so that it is half a turn counterclockwise from the point at which the PFPD goes out of tick-tock.

5. In the Gate Parameters screen, set the integration gate's start and stop times (recommend 6 ms and 24.9 ms respectively) to optimize sulfur detectivity. If the gas flow rates are properly set, the sulfur emission extends to 24 ms or beyond for 2-mm combustors (Figure 6.5).

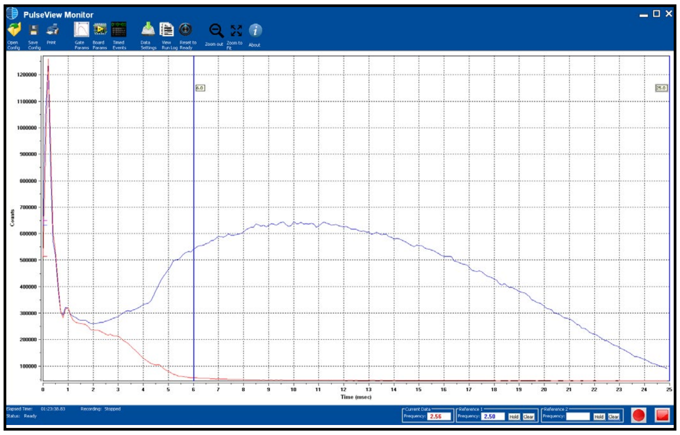


Figure 6.5. Sulfur Emission and Gate Settings for Maximizing Sulfur Response Using a 2-mm Combustor

6. With a 3-mm combustor, the sulfur emission extends to around 20 ms, and the emission appearance differs (Figure 6.6).

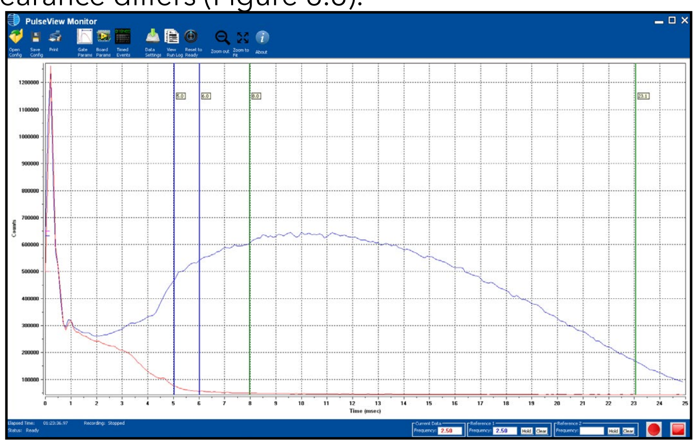


Figure 6.6. Sulfur Emission and Gate Settings for Maximizing Sulfur Response Using a 3-mm Combustor

For high sulfur concentrations, reduce the sulfur concentration in the combustor and maximize the PFPD's detectivity for sulfur using the following steps:

1. Decrease sensitivity by decreasing the PMT voltage and range settings in PulseView. If the emission starts to collapse (i.e., quench) when using the 2-mm combustor, replace it with a 3-mm combustor.

2. Set the detector temperature to 250 °C. Using higher temperatures for some applications if needed may result in a small sensitivity decrease.

3. Set the combustor gas composition to the optimum H_2-rich mixture.

4. Set the fine adjust needle valve so that it is half a turn counterclockwise from the point at which the PFPD goes out of tick-tock.

Optimizing Selectivity

Increasing selectivity for a specific analyte greatly simplifies chromatographic analysis PulseView Monitor lets operators specify gate settings that minimize or eliminate interferences from other analytes, increasing selectivity for the analyte of interest. Enhance selectivity for analytes with overlapping chemiluminescent lifetimes using the software's dual gate capability (see Chapter 14, "Dual Gate Capability and Alpha Calculation" for a detailed discussion). The dual gate capability can be particularly useful for maximizing sulfur-to-carbon, phosphorus-to-carbon, sulfur-to-phosphorus, and phosphorus-to-sulfur selectivity. Figure 6.7 shows possible gate settings for selecting phosphorus and sulfur.

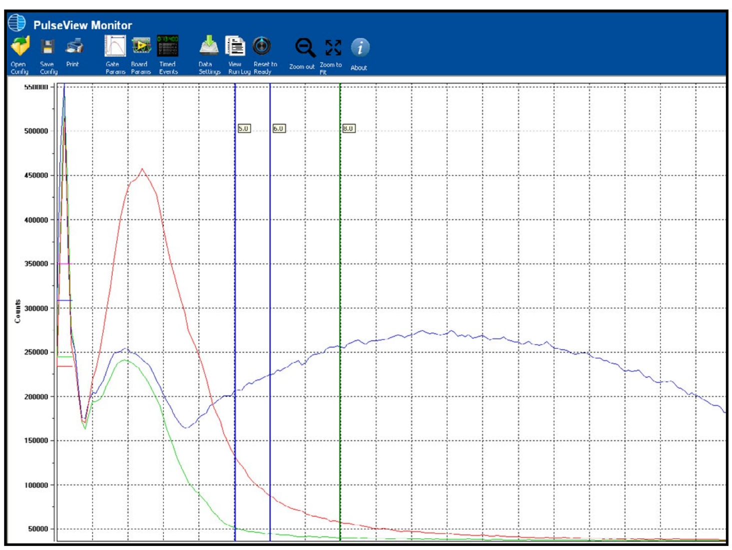


Figure 6.7. C2*, OH*, CH*, Phosphorus, and Sulfur Emissions with Gate Settings for Phosphorus and Sulfur

The phosphorus emission was obtained using 400 V PMT voltage, and the sulfur emission was obtained using 600 V PMT voltage.

Maximizing Phosphorus-to-Carbon Selectivity

Maximize the PFPD's selectivity for phosphorus over carbon using the following steps:

1. Use an optical filter in the PMT assembly that is optimal for detecting phosphorus (see Chapter 12, "Photomultiplier Tube and Filter Configurations for Various Element Detection"). The GG-495 optical filter is recommended for optimum phosphorus performance.

2. Optimize for phosphorus response as described in "Maximizing Phosphorus Response" in this chapter.

3. Delay the gate A start time relative to that shown in Figure 6.4 to increase selectivity between the phosphorus and hydrocarbon emissions (i.e., increase the gate A **Start Time** in the Gate Parameters screen to 4–6 ms or more), minimizing hydrocarbon and phosphorus response overlap.

Maximizing Sulfur-to-Carbon Selectivity

Maximize the PFPD's selectivity for sulfur over carbon using the following steps:

1. Ensure the carbon concentration is not so high that it quenches the sulfur response. Quenching can be observed by a sulfur emission collapse (i.e., reduction in emission lifetime) during coelution with hydrocarbon concentrations. If the carbon flow rate exceeds 10–100 ng/sec, reduce it by splitting the sample.

2. Use an optical filter in the PMT assembly that is optimal for detecting sulfur (see Chapter 12, "Photomultiplier Tube and Filter Configurations for Various Element Detection"). The BG-12 Optical filter is recommended for optimum sulfur performance.

3. Optimize the PFPD for sulfur response as described in Maximizing Sulfur Response in this chapter.

4. Delay the gate start time relative to that shown in Figure 6.5 (i.e., increase the gate A **Start Time** in the Gate Parameters screen to 6–8 ms), minimizing the hydrocarbon and sulfur response overlap. Due to the sulfur emission's long time domain, a correctly delayed gate virtually eliminates carbon interferences without using dual gate techniques.

Maximizing Phosphorus-to-Sulfur Selectivity

Use an optical filter in the PMT assembly that is optimal for detecting phosphorus and minimizing the sulfur response (see Chapter 15, "Noise Sources and Output Signal Optimization"). Using a GG-495 filter (PN 282947) greatly reduces any sulfur response. Even with another filter in place that allows both phosphorus and sulfur detection, the following steps still increase phosphorus selectivity.

1. Because HPO emission optimizes at a more oxygen-rich flame than sulfur, set the phosphorus H_2:Air 1 flow ratio at the phosphorus optimal detection flow rate.

2. Optimize the PFPD for phosphorus response as described in “Maximizing Phosphorus Response” in this chapter. Because phosphorus emits earlier than sulfur, an earlier gate termination improves the phosphorus-to-sulfur selectivity. Use a 3-mm combustor.

3. Set the PFPD to 300 °C.

4. Activate the PFPD’s dual gate capability to enhance selectivity between phosphorus and sulfur without large adjustments to gate settings for optimal phosphorus detectivity (see Chapter 14, “Dual Gate Capability and Alpha Calculation” for further details).

Maximizing Sulfur-to-Phosphorus Selectivity

Use an optical filter in the PMT assembly that is optimal for detecting sulfur (see Chapter 12, “Photomultiplier Tube and Filter Configurations for Various Element Detection”). Using a BG-12 filter (PN 282947) greatly reduces any phosphorus response. Even with another filter in place that allows both phosphorus and sulfur detection, the following steps still increase sulfur selectivity.

1. Set the H_2:Air 1 flow ratio at the sulfur detection optimal flow rate.

2. Optimize the PFPD for sulfur sensitivity as described in “Maximizing Sulfur Response” in this chapter. Because sulfur emits later than phosphorus, increased gate delay improves the sulfur-to-phosphorus selectivity. Use a 2-mm combustor.

3. Set the PFPD to 220 °C.

Since delaying the gate reduces detectivity, activate the PFPD’s dual gate capability to enhance selectivity without large adjustments to gate settings for optimal sulfur detectivity. This enables the operator to adjust for phosphorus responses within the detectivity-optimized time domain of Gate A (see Chapter 13, “Defining Detectivity” for further details).

Minimizing Quenching

1. Inject the smallest possible sample size that provides sufficient sensitivity. Use the3-mm combustor.

2. Increase the Air 1 flow rate by 1–2 mL/minutes or until the sulfur response is reduced by a factor of about two (or at least 1.2).

3. Set the **Gate Start** at 5 ms and the **Gate Stop** at 12–15 ms. Quenching mostly affects the longer delayed emission.

4. Use a semipolar column that separates critical pairs such as thiophene and benzene or methylthiophene and toluene. This column needs a thick film to reduce column saturation and maintain good separation. For gasoline analysis, use an Rtx®-35 column with a 1-µm film and 0.25-mm I.D. (or equivalent). For gas analysis such as COS in propylene, use the silica PLOT column from Chrompack with a 4-µm PLOT film and 32-mm I.D.

Detecting Phosphorus and Sulfur Simultaneously

1. Install a UV34 optical filter (PN 310623) (see Maximizing Sulfur Response).

2. Select which element's sensitivity is more important to determine the combustor and optimal gas flow rate. For information on choosing the appropriate combustor, contact the OI Analytical Technical Support Department at 800-336-1911 or 979-690-1711.

3. Install the appropriate combustor (see Chapter 4, "Installing the Combustor and Reassembling the PFPD") and set the optimal gas flow rate (see "Optimizing the Combustor Flow (Sulfur)" in this chapter) for the select element sensitivity. Note that the 2-mm combustor reduces or increases the detectivity of the element by less than a factor of two compared with the 3-mm combustor.

4. Using the GC's control panel, set the PFPD base temperature to 300 °C. The higher temperature favors phosphorus detectivity, but it is needed for equimolar phosphorus response. A lower temperature increases sulfur detectivity.

5. From PulseView Monitor, set the sulfur gate start to 6 ms and the gate stop to 24.9 ms. Set the phosphorus gate to start at 5 msec and stop at 15 msec.

6. Use dual gate subtraction to optimize mutual sulfur and phosphorus selectively (see Chapter 14).

Testing PFPD Performance

Test the PFPD's performance only after optimizing for the desired detectivity and selectivity. This presumes that suitable filters are installed, the PMT voltage is set correctly for detecting the analyte, the H_2:Air 1 flow ratio and integration gate settings are optimized, and the correct detection mode is specified and selected in PulseView Monitor. Use the following additional measures for correct PFPD operation.

1. Configure the data integration system to display a detector baseline with sufficient resolution to measure PFPD noise. Collect data for a time that is sufficient to accurately measure the detector noise and peak-to-peak noise level (see Chapter 15, "Noise Sources and Output Signal Optimization") or the PFPD Quick Reference Guide.

2. Use the standard test solution included in the PFPD startup kit or other suitable text mix to calculate detectivity.

3. Inject the standard test solution into the GC injector and simultaneously start the data integration system. Note the amount of sample injected into the column and calculate the amount of sulfur or phosphorus for each analyte in the sample.

4. Ensure the multiplier parameters are set to provide sufficient noise resolution. The PMT voltage may also require some adjustment (default is 600 V) so that the output signal does not exceed the PFPD's signal handling capacity. Alternatively, if the output signal is low, increase the PMT voltage or the multiplier to increase the signal. See step 14 of "Starting the PFPD System" in this chapter for information on optimizing the PMT voltage.

5. Monitor the chemiluminescent emission with PulseView Monitor to ensure the emission does not exceed the PFPD's maximum signal handling capacity (i.e., the emission peak remains below 16,800,000 counts in PulseView Monitor). If this occurs, reduce the sample concentration or reduce the PMT setting in PulseView Monitor, and rerun the test. The upper limit of 16,700,000 counts occurs only when the emission levels within the gate are saturated. Saturation normally begins when only a portion of the signal under the gate is saturated, or the multiplier is set too high. This effect is observes as a truncated peak (flat top).

6. Measure the peak height and width at half height (or width at quarter height for sulfur in the standard, nonsquare root mode) for each peak resulting from the chromatographic output. Calculate detectivity for each analyte and determine if the detectivity and or selectivity is sufficient for the intended application. (See Chapter 13 Defining Detectivity, for calculating detectivity for sulfur and phosphorus.)

Figure 6.8 provides chromatographic examples of a standard test for sulfur run at OI Analytical. The following specifications were used to obtain this output.

- The carrier gas is helium, and the flow rate is 1.2 mL/min.
- Column specifications are: HP-5 5% phenyl methyl siloxane, 30.0 m length, 0.32 mm diameter, and 0.25-µm film thickness.
- Temperatures settings are listed below:
  - Splitless isothermal injector set at 250 °C,
  - GC oven is programmed as follows:
    - Initial temperature set at 60 °C, hold for 0.50 min, Ramp 25 °C/min to 180 °C,
    - Ramp at 8 °C/min to 260 °C,
    - Ramp at 25 °C/min to 300 °C, hold for 2.00 min,
  - PFPD temperature set at 250 °C.

The test standard contains 500 pg/µL thimet, 1,200 pg/µL diazinon, and 1,500 pg/µL chorpyrifos. Figure 6.8 shows a chromatogram of the quadratic response output for sulfur, obtained using the conditions described above.

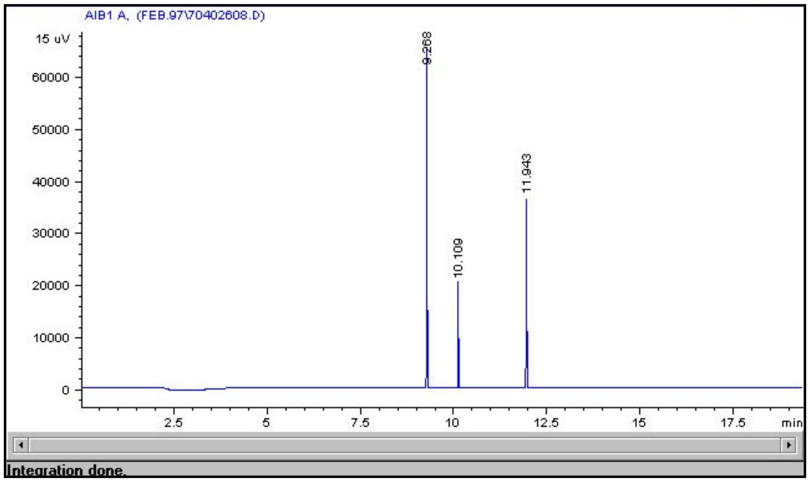


Figure 6.8. Chromatogram of the OI Analytical Test Standard (Sulfur Mode)

Recording PulseView Monitor Files for Viewing with PulseView Analyzer

Record PulseView Monitor data in a format that can be viewed and manipulated in PulseView Analyzer. PulseView Analyze is a standard software package for the 5383 PFPD. Use this program to reprocess (post run) portions of or an entire chromatographic run under different PFPD operational parameters without rerunning the chromatographic analysis.

1. Define the directory and file name for the recorded PulseView Monitor files.

   - Click the **Data Settings** button on the button bar of the PulseView Monitor Main screen.
   - Select a location and name for the PulseView Monitor files.
   - Click **Save**. The new file name appears in the File Name section of the PulseView Monitor Main screen. The file name is followed by a three-digit extension, such as 001. The extension increments by one for each additional file that is recorded (e.g., 002, 003, 004, etc.).

2. Click **Record** at the beginning of a GC run to record the data manually (without the remote start cable). To stop recording, click **Stop Record**.
   To record the data using the remote start cable, complete the following steps:
   a. Verify the remote start cable is installed correctly (see Chapter 3, "Installing the Model 5383 PFPD Detector Assembly onto the Shimadzu GC-2014").
   b. Click the **Timed Events** button to access the Timed Events screen.
   c. Enter in the start time, select **Record** from the Event list box, and enter in the stop time (relative to the start of a GC run). Click **Add**.
   d. Click **OK** to save the timed events. PulseView Monitor is now programed to begin recording at the specified times. See Chapter 4, "Timed Events Screen" for more information.

NOTE: PulseView Monitor files can become large in size very quickly. Go to the Utilities menu and select Set Preferences to minimize the size of the PulseView Monitor files. Set the maximum data file size for a run. The data file automatically closes when the maximum limit for the data file is met.

Chapter 7 Maintenance

This chapter describes the 5383 PFPD maintenance. Properly storing and handling detector components helps ensure reliable PFPD operation. Never handle combustor tools or forceps parts that touch internal detector parts. Use clean laboratory tissues, the Teflon extractor device, or Teflon-coated forceps to remove or replace internal detector parts. Place removed parts on a clean, lint-free laboratory tissue or in a clean glass beaker.

Changing the Combustor

Replace the combustor either when a combustor with another internal diameter is needed or when the carbon emission (second emission) begins tailing excessively (i.e., beyond six milliseconds from the trigger point). When conditioning a column attached to the PFPD, install an old, dirty combustor, so as not to contaminate a clean combustor.

This section describes how to replace the combustor and associated parts housed within the detector body, and how to inspect the top of the GC column. See Figure 7.1 for part names and numbers.

NOTE: Before changing the combustor, bake the PFPD overnight at 350 °C. Observe the carbon emission to see if the combustor is clean. In most cases, this procedure cleans the combustor sufficiently.

NOTE: The combustor is sensitive to dust. If the baking process fails to clean the combustor, replace it.

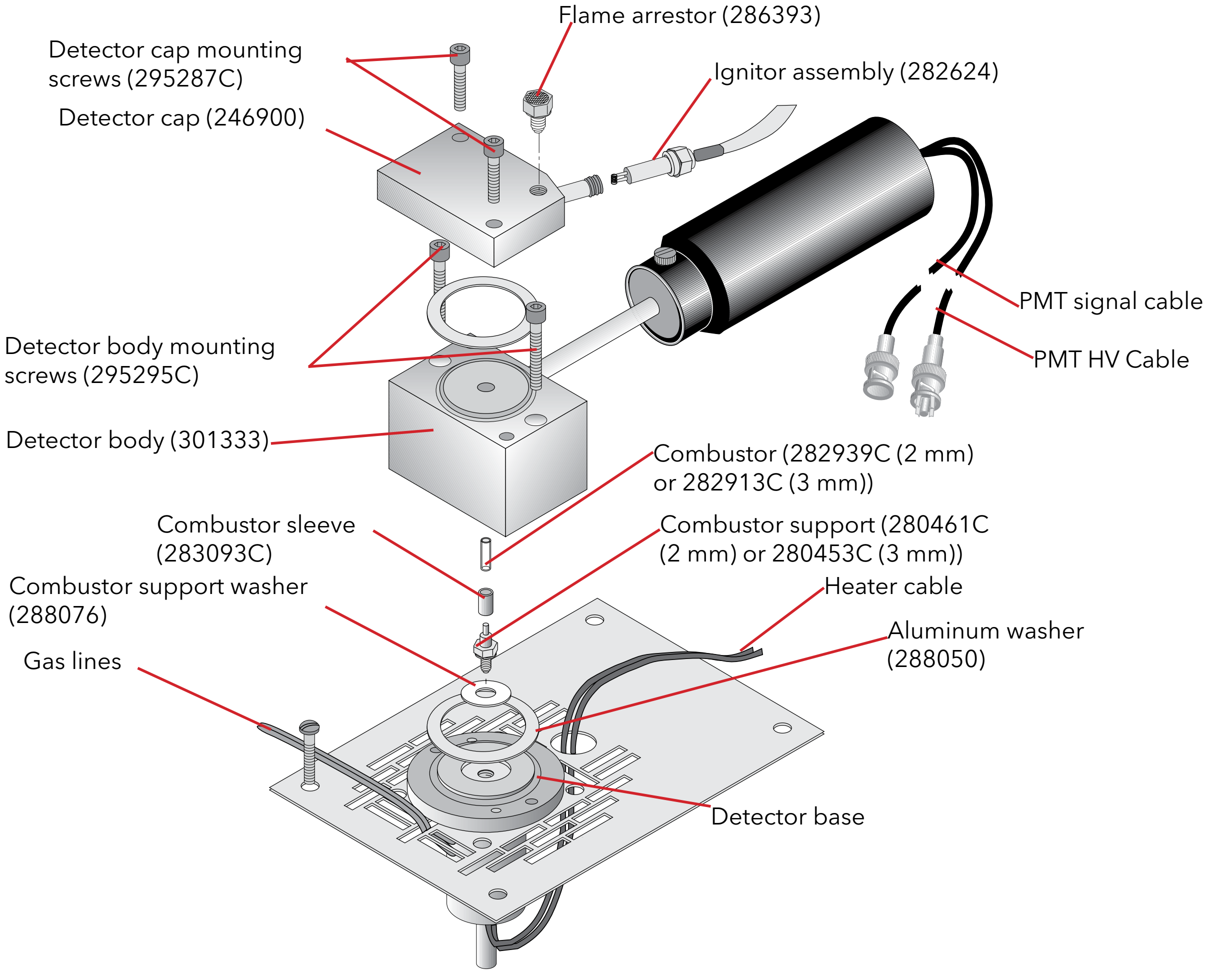


Figure 7.1. 5383 PFPD Assembly

Removing the Installed Combustor

WARNING: Handling the PFPD while still hot can cause serious burns.

CAUTION: Due to high temperatures, the mounting screws may become increasingly difficult to remove. If experiencing any resistance in removing them, replace them with new screws.

1. Set the PMT voltage and ignitor current to zero using PulseView Monitor.

2. Turn the detector controller off, and disconnect the PMT HV cable from the back of the detector controller. Allow the PFPD to cool to room temperature.

3. Remove the detector cap mounting screws (PN 295287C) using a 7/64" Allen wrench. Place the screws on a clean laboratory tissue.

4. Lift the detector cap (PN 246900) off the detector body (PN 301333) and place it upside down onto a clean laboratory tissue. Remove and dispose of the large aluminum washer (PN 288050) loosely attached to either the bottom of the detector cap or the top of the detector body.

5. Using the appropriate combustor extractor tool (PN 280719 for the 2-mm combustor; PN 280727 for the 3-mm combustor) (Figure 4.36) and Teflon-coated forceps, carefully remove the combustor from the detector body. Invert the combustor extractor tool, immediately reinsert it into its protective sheath, and stand it on end. Place it where it cannot be accidentally knocked over.

6. After the combustor is placed within the protective sheath, carefully examine the combustor for signs of contamination cracks, or chips. If the combustor does not appear damaged or contaminated, place it in the plastic receptacle in which it was supplied or in a clean, sealable plastic bag, and store it in a safe place. If only changing the combustor, go to “Installing the Combustor” in this chapter.

Examining the Column Tip

CAUTION: Due to high temperatures, the mounting screws may become increasingly difficult to remove. If experiencing any resistance in removing them, replace them with new screws.

1. Verify the combustor is removed.

2. Remove the detector body mounting screws (PN 295295C) using a 7/64" Allen wrench. Place the screws on a clean laboratory tissue.

3. Gently lift the detector body straight up and off of the detector base, taking care not to crush the column extending beyond the top of the combustor support. Place the detector body on its side on a clean laboratory tissue. Remove and dispose of the large aluminum seal washer (PN 288050) from either the bottom of the detector body or the top of the detector base.

4. Examine the top of the column extending beyond the top of the combustor support. It should be nearly transparent. The polyimide coating should be burned away leaving only a clean section of fused silica tubing exposed. If the polyimide coating is not completely burned away, the flame is not terminating at the bottom of the combustor. Increase the Air 1 flow rate (or decrease the H_2 flow) in subsequent PFPD operations. If poor performance is the reason for changing the combustor, retest the detector’s performance by adjusting the gas flows, which may eliminate the need to replace the combustor. If so, go to “Installing the Detector Body” in this chapter.

Replacing the Combustor Support

CAUTION: Crushing the capillary tube tip during detector body installation impairs chromatographic performance. Reinstall the column if this occurs.

1. Remove the combustor sleeve and store it in a clean plastic bag.

2. Remove the combustor support (PN 280461C for the 2-mm combustor; PN 280453C for the 3-mm combustor) from the detector base using a clean 3/8" wrench. Place the combustor support onto a clean laboratory tissue or store in a clean, sealable plastic bag.

3. Loosen the column nut (PN 223057) in the GC oven and pull the column down.

4. Replace the aluminum combustor support washer (PN 288076) from the counter bore in the center of the detector base using clean forceps.

5. Place the desired combustor support into the detector base using a clean laboratory tissue. Finger-tighten and tighten an additional 1/8–1/4 turn, using a clean 3/8" wrench.

6. Reinstall the combustor sleeve.

7. Carefully slide the column back up through the column nut and into the detector base until it protrudes beyond the top of the combustor support. (Refer to the appropriate GC manuals for general instructions on how to install a capillary column into a detector.)

8. Using a magnifying lens, see if the column needs trimming. If so, trim the column using a column scribe and verify a square cut is achieved. Carefully pull the column back down until it is 0.5–1.5 mm above the top of the combustor support. Tighten the column nut. Check and verify the column’s position above the combustor support is still correct.

Installing the Detector Body

CAUTION: Due to high temperatures, the mounting screws may become increasingly difficult to remove. If experiencing any resistance in removing them, replace them with new screws.

1. Place a new aluminum washer (PN 288050) on the detector base using clean metal forceps.

2. Look through the center of the detector body and guide the detector body over the combustor support, keeping the combustor support centered in the detector body.

3. Secure the detector body to the detector base using the two mounting screws (PN 295295C), keeping any debris from falling into the detector body through the holes. Alternate tightening the screws using a 7/64" Allen wrench until the detector body is touching the detector base.

Installing the Combustor

CAUTION: Due to high temperatures, the mounting screws may become increasingly difficult to remove. If experiencing any resistance in removing them, replace them with new screws.

1. Carefully place the combustor (PN 282939C for the 2-mm combustor; PN 282913C for the 3-mm combustor) into the center hole of the detector body using the appropriate combustor extractor tool (PN 280719 for the 2-mm combustor; PN 280727 for the 3-mm combustor).

2. Lower the combustor vertically into the hole. If the combustor sticks to the combustor extractor tool, use clean, Teflon-coated forceps to gently pry the combustor off of the extractor tool and into the center hole of the detector body. The combustor is properly installed when the top of it sits approximately 0.05" below the surface of the detector body.

3. Place a new aluminum washer (PN 288050) onto the detector body.

4. Install the detector cap on the top of the detector body, centering the large aluminum seal washer with the counter bore in the ignitor cap.

5. Secure the detector cap with the two detector cap mounting screws (PN 295287C).

6. Alternately tighten the two screws using an Allen wrench. A gap between the detector cap and the detector body exists, which should be approximately 0.02" (0.5 mm) the same all around.

7. Connect the PMT HV cable to the detector controller. Verify the signal cables are routed correctly and that they are connected to the detector controller. Verify the PMT housing is properly seated on the PMT mount located on the detector body.

8. Turn on the detector controller. Using PulseView Monitor, set the PMT voltage and ignitor currents to the desired set value.

Testing and Replacing the Ignitor Assembly

Test and replace the ignitor assembly (PN 282624) if the PFPD does not ignite when appropriate gas flows and the ignitor current are set. Figure 7.2 shows the part of the ignitor assembly that is housed in the detector cap.

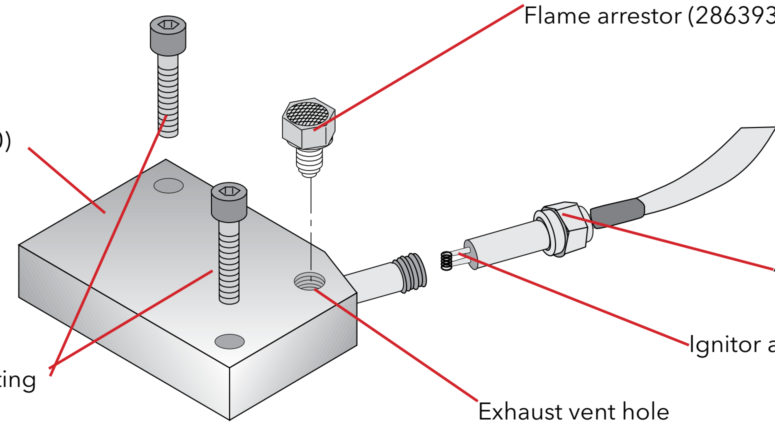


Figure 7.2. Ignitor Assembly

WARNING: Handling the PFPD while still hot can cause serious burns.

1. Remove the flame arrestor. Look down through the vent and verify the ignitor coil is not glowing when the ignitor current is off.

2. Set the ignitor current to zero.

3. Turn off power to the detector controller and unplug the power cable.

4. Disconnect the ignitor cable from the detector controller. Allow the detector cap to cool to room temperature.

5. Determine if the ignitor is open using the ohm scale on an ohm-volt meter. Measure the resistance between the leads on the ignitor cable by inserting the probes into the three-pin connector that connects to the detector controller. If it is open and showing very large resistance, replace the ignitor assembly.

6. Measure the resistance using the ohm-volt meter between one lead and the top of the detector cap to see if it is shorted to the detector cap. If it appears shorted, look through the exhaust vent hole on the detector cap and verify the ignitor is centered in the exhaust vent hole. If it is not centered, the ignitor may be shorting to the wall of the ignitor cap. If possible, loosen the ignitor assembly to center the ignitor in the exhaust vent hole. Recheck to see if it is still shorting. If it is still shorting out, replace the ignitor assembly.

7. Detach the ignitor assembly from the detector cap using a 7/16" wrench to loosen the attachment nut.

8. Install the ignitor, tightening the attachment nut until the coil can be seen centered in the exhaust vent hole. Verify the ignitor is not shorting to the detector cap and that the ignitor assembly is not open.

9. Reconnect the ignitor cable to the detector controller.

10. Turn on the detector controller. Reset the PMT voltage and ignitor current to the desired set value using PulseView Monitor.

Replacing the Optical Components

CAUTION: Never use bare hands when installing the PFPD optics. Never handle tool parts used to remove or replace internal parts of the detector. Always use clean powder-free latex gloves and Teflon-coated forceps to remove and replace internal optical parts. Place optical parts on a clean, lint-free laboratory tissue or in a clean glass beaker, unless otherwise stated.

The three main components of the PFPD's photomultiplier assembly that can be replaced are the PMT, the optical filter, and the light pipe (Figure 7.3).

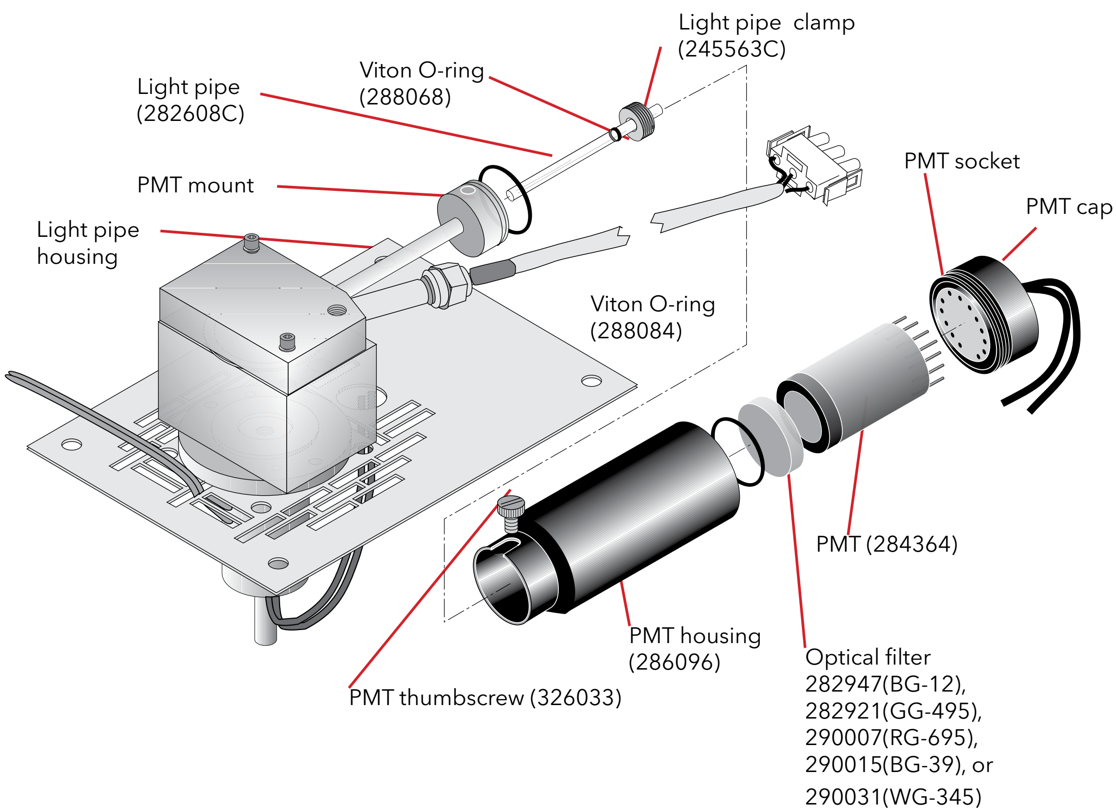


Figure 7.3. Photomultiplier Tube (PMT) Assembly

Replacing the Photomultiplier Tube

WARNING: Handling the PFPD while still hot can cause serious burns.

CAUTION: The PMT optical parts are easily damaged and may need replacing if they fall out of the PMT housing.

CAUTION: Do not use excessive force when tightening the PMT housing together. PMTs can be easily damaged by overtightening the housing assembly.

CAUTION: Check for light leaks if the PMT voltage appears unable to accept any value other than zero volts.

The PMT should not need frequent replacing if it is handled correctly. If the output signal begins to deteriorate, increase the PMT voltage. If the required PMT voltage becomes excessively high or if the type of PMT needs changing, use the following procedure

1. Set the PMT voltage and ignitor current to zero using PulseView Monitor. Disconnect the PMT HV cable and the PMT signal cable from the detector controller.

2. Turn off the detector controller and allow the detector to cool to room temperature.

3. Detach the PMT housing assembly (PN 286096) from the PMT mounting by loosening the PMT thumbscrew (PN 326033) and gently pulling off the housing. Wiggle the housing back and forth if necessary to remove it. Open the PMT housing assembly by unscrewing the PMT housing counterclockwise from the cap (Figure 6.3).

4. Place the PMT housing assembly onto a clean, lint-free laboratory tissue, ensuring none of the contents fall out.

5. Carefully detach the PMT (PN 284364, 290049, or 290106) from the PMT socket and place it onto a clean, lint-free laboratory tissue or in an optical storage container.

6. Insert the new PMT into the keyed PMT socket. Reinsert the PMT into the PMT housing body. Screw the cap with attached PMT back onto the PMT housing body. If the filter rattles when the cap is fully screwed on, use an additional Viton O-ring (PN 288084) to fill excess space between the PMT and the filter. If this is the case, remove the PMT again, install an additional Viton O-ring, and reassemble the PMT assembly.

7. Verify the PMT Viton O-ring, which causes the PMT housing assembly to fit snugly onto the PMT mounting, is in place and not damaged. Replace if necessary. Twist the PMT housing assembly back and forth onto the PMT mounting and reattach it using the thumbscrew.

8. Route the PMT cables to avoid high thermal zones, sharp edges, or places where the cable could be pinched or sheared.

9. Connect the PMT signal cable and the PMT HV cable to their respective connectors on the back of the detector controller.

10. Reset the PMT voltage (e.g., nominally 600 V) using PulseView Monitor.

11. Optimize the PMT voltage. See step 10 in Chapter 6 Starting the PFPD System.

Replacing the Optical Filter(s)

WARNING: Handling the PFPD while still hot can cause serious burns.

CAUTION: Touching the face of the PMT tube can damage the optical sensitivity of the PFPD.

CAUTION: Do not use excessive force when tightening the PMT housing together. PMTs can be easily damaged by overtightening the housing assembly.

If changing the PFPD's operational mode, the optical filter may have replacing in the PMT housing assembly (Figure 7.3). Chapter 12, "Photomultiplier Tube and Filter Configurations for Various Element Detection" provides a list of suitable filters for different modes. Use the following procedure if the optical filter(s) needs replacing:

1. Set the PMT voltage to zero using PulseView Monitor. Disconnect the PMT HV and the PMT signal cables from the back of the Detector Controller.

2. Turn off the Detector Controller and allow the PFPD to cool to room temperature.

3. Detach the PMT housing assembly (PN 286096) from the PMT mounting by loosening the PMT thumbscrew (PN 326033) and gently pull off the housing. Wiggle the housing back and forth, if necessary, to remove it. Open the PMT housing assembly by unscrewing the PMT housing counterclockwise from the cap (Figure 7.3).

4. Place the PMT housing assembly onto a clean, lint-free laboratory tissue, ensuring none of the contents fall out.

5. Remove the existing filter(s) and Viton O-ring from the PMT housing and place them onto a clean laboratory tissue or other optical storage container.

6. Install the Viton O-ring and the replacement filter in the PMT housing.

7. Verify the PMT is not broken and is properly mounted in its socket. Carefully screw the PMT housing cap back onto the body of the PMT housing. Lightly tighten the PMT cap until the filter(s) does not rattle or move easily. If the filter(s) is loose when the cap is fully tightened, remove the PMT again, install an additional Viton O-ring to take up the excess space, and reassemble the PMT housing.

8. Verify the PMT Viton O-ring, which causes the PMT housing assembly to fit snugly onto the PMT mounting, is in place and not damaged. Replace if necessary. Twist the PMT housing back onto the PMT mounting, and reattach it using the thumbscrew.

9. Route the PMT cables to avoid high temperature zones, sharp edges, or places where the cable could be pinched or sheared.

10. Connect the PMT signal cable and the PMT HV cable to their respective connectors at the back of the detector controller.

11. Reset the PMT voltage (e.g., nominally 600 V) using PulseView Monitor.

12. Optimize the PMT voltage. See step 10 in Chapter 6 Starting the PFPD System.

Replacing the Light Pipe

WARNING: Handling the PFPD while still hot can cause serious burns.

CAUTION: Touching the face of the PMT tube can damage the optical sensitivity of the PFPD.

CAUTION: Do not scratch the protruding end of the light pipe with the screwdriver when tightening the light pipe clamp.

The light pipe does not need replacing under normal usage conditions. If it is cracked or broken, use the following procedure to replace it.

1. Set the PMT voltage to zero using PulseView Monitor. Disconnect the PMT HV and the PMT signal cables from the back of the detector controller.

2. Turn off the detector controller and allow the PFPD to cool to room temperature.

3. Detach the PMT housing assembly (PN 286096) from the PMT mounting by loosening the PMT thumbscrew (PN 326033) (Figure 7.3). Place it where it cannot accidentally drop or roll.

4. Unscrew the light pipe clamp (PN 245563C) in the end of the PMT mounting using a screwdriver or other appropriate tool, and remove the light pipe from the detector body using Teflon-coated forceps.

   If light pipe fragments are found from a chipped, cracked, or broken light pipe, remove the detector cap, combustor, and detector body, and discard the glass fragments in a safe manner to prevent injury. Using a cotton swab dipped in methanol or isopropanol, wipe the light pipe tube protruding from inside the detector body to remove all glass fragments. Discard the cotton swab in an appropriate waste container.

5. Reassemble the detector body, combustor, and detector cap after ensuring there are no glass fragments in the combustor chamber, inside the detector body, or in the detector base.

6. Remove the new light pipe from its protective cover using a clean laboratory tissue.
7. Examine the light pipe for smudges, cracks, chips, or other deformations. If the light pipe has mechanical imperfections, install another light pipe. If smudges (fingerprints, dirt, or oil) appear on its surface, dampen another clean laboratory tissue and lightly wipe the light pipe.

8. Install a Viton O-ring (PN 288068) at least 1.5 cm from one end of the light pipe using another clean laboratory tissue.

9. Install the other end of the light pipe into the light pipe housing. Thread the light pipe clamp into the PMT mounting attached to the end of the light pipe housing.

10. Lightly tighten the light pipe clamp slightly to compress the Viton O-ring using a narrow screwdriver or other appropriate tool.

11. Push the light pipe into the light pipe housing until it is flush with the edge of the PMT mounting using a clean laboratory tissue. If the light pipe does not slide in easily, loosen the light pipe clamp and try again. Tighten the light pipe clamp an additional 1/4–1/2 turn. Ensure the light pipe does not extend more than 1.5 mm beyond the edge of the light pipe mount. Greater protrusion results in damage to the light pipe, filter(s), or PMT.

12. Verify the PMT Viton O-ring, which causes the PMT housing assembly to fit snugly onto the PMT mounting, is in place and not damaged. Replace if necessary. Twist the PMT housing assembly back and forth onto the PMT mounting and attach it using the thumbscrew.

13. Route the PMT cables to avoid high thermal zones, sharp edges, or places where they could be pinched or sheared.

14. Connect the PMT signal cable and the PMT HV cable to their respective connectors at the back of the detector controller.

15. Reset the PMT voltage to its appropriate voltage (e.g., nominally 600 V) using PulseView Monitor.

16. Optimize the PMT voltage.

Cleaning the Model 5383 PFPD

Clean the various components, tools, and optics of the 5383 PFPD using the following procedures.

Cleaning Optical Components

Optical components consist of the light pipe, optical filter, and the PMT window (face of the PMT). Dampen a clean, lint-free laboratory tissue or optical tissue with methanol or isopropanol and lightly wipe the surfaces to remove fingerprints or other contaminants.

NOTE: Handle the components with clean latex gloves, optical tissues, or lint-free laboratory tissues. Store them in their protective containers.

Cleaning Combustors

In most cases, the combustor cannot be cleaned back to original performance levels. If the combustors are not performing properly, replace them. Store the combustors in their protective containers.

Cleaning the Detector Cap, Body, Base, and Combustor Support

CAUTION: Do not exceed 420 °C, the maximum recommended temperature for the PFPD.

OI Analytical rigorously cleans and bakes these parts prior to shipping to remove all traces of machining oils. Remove normal contamination due to handling, dust, or contact with dirty surfaces using organic solvents such as methanol, isopropanol, hexane, and acetone.

1. Pass solvents through all sections of tubing to ensure the contaminants are not simply displaced into normally inaccessible spaces.

2. Bake off the solvents at a sufficiently high temperature (e.g., 350 °C) to remove the solvent and contaminants possibly in the solvent.

3. If reusing the cleaned detector components, bake the components after reassembling the PFPD using a staged heating process. The stage process used by OI Analytical includes setting the initial detector temperature to 200 °C, increasing it to 350 °C after an hour, and decreasing it back to 200 °C after another hour.

4. Allow all parts to cool to room temperature prior to storage or installation.

Cleaning Sealing Washers

1. Place the sealing washers in a clean beaker with methanol or isopropanol and sonicate.

2. Remove washers from the beaker using clean metal forceps.

3. Put the washers into a clean beaker and bake at 70 °C for 30 minutes.

4. Allow the washers to cool to room temperature and store them in a protective container.

Cleaning O-rings, Extractor Tools, and Teflon Forceps

1. Place the O-rings, extractor tools, or Teflon-coated forceps in a clean beaker with methanol or isopropanol and sonicate.

2. Remove the components from the beaker using clean Teflon forceps and place them in a clean beaker. Bake at 150 °C for four hours.

3. Let the components cool to room temperature and store them in a clean protective container.

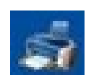
Chapter 8 Troubleshooting

The following lists the most common possible problems when using the PFPD, along with their most probable causes and corresponding corrective actions. Each problem potentially causes more than one symptom. The probable causes of each symptom are listed in order of increasing severity. This manual discusses each corrective action in previous chapters, under either an installation, operation, or maintenance procedure.

Before using this guide, become thoroughly familiar with the operation and maintenance information contained in previous chapters.

If none of the following corrective actions correct the problem(s) being experienced, contact OI Analytical's Technical Support Department at (800) 336-1911 or (979) 690-1711 with the following three printouts available:

1. PulseView Monitor screen showing a peak emission signal: To capture a peak response during a chromatographic run, activate one of the two hold buttons on the right of the Main screen at the moment that the emission signal reaches its maximum value. To save the screen:
   - Press **Alt+Print Screen** on the PC's keyboard
   - Open the Paint program (**Start** > **Accessories** > **Paint** from the status bar at the bottom of the screen).
   - Go to the **Edit** menu and select **Paste**.
   - Save the captured screen to a file, and print out a copy of the captured screen.

2. PulseView Monitor parameter values: To obtain this, click the  button at the top of the main screen

3. Chromatogram: If a PC-based program such as Agilent ChemStation is being used to generate the chromatogram, obtain a printout using the same screen capture sequence described for capturing a PulseView Monitor screen.

Symptom	Probable Cause	Corrective Action
No signal	PC's USB-to-485 cable not connected to the detector controller	Check the USB cable connection to the PC and detector controller.
	Incorrect Com port assigned	Reassign the Com port.
Flame ignition problems	Gas flows too low or not set correctly	Verify the carrier, H_2, and air pressure regulators are set correctly and that gas is supplied to the pneumatics module.
	EPC controllers off	Turn the EPC controllers on.
	Carrier gas flow rate too high	Decrease the carrier gas flow rate (if possible), increase Air 1 to increase the flame temperature in the combustor, or use a 3-mm combustor.
	Detector leaking gas from washers or other seals	Check for gas leaks around the aluminum sealing washers. Check column connections and verify the column's seal is secure and is not broken.
	Ignitor broken or shorted	Replace the ignitor. See Chapter 7, "Testing and Replacing the Ignitor Assembly".
	Ignitor current set incorrectly	Verify the ignitor current is set at 2.8 A and optimize Air 1 flow. Increase the ignitor setting to 3.3 A until a regular pulse is obtained. Reset it to 2.8 A.
	Cold detector body	Heat the PFPD to 220–300 °C.
Flame propagation problems	Inconsistent flame propagation into the combustor (tick-tock)	Set the GC oven to the highest temperature that the analysis requires. Then adjust the needle valve counterclockwise (open) to bring it out of tick-tock.
	Incomplete flame propagation through the combustor	Examine the section of the column that extends above the combustor support. If the column's polyimide coating has not been cleanly burnt off, add more Air 1 (or reduce the H_2 flow).

PFPD quits pulsing when solvent is injected and does not restart after solvent elutes	Ignitor current set too low	Increase the ignitor current to a higher value (maximum is 3.3 A) and reset it to 2.8 A after a regular pulse is achieved.
	H_2 set too high	Decrease the H_2:air ratio.
Noisy detector	PMT seal not properly made	Secure the PMT housing to the PMT mounting on the detector body with the supplied thumbscrew.
	Cracked or deformed PMT housing	Replace the PMT housing.
	Filter(s) incorrectly installed	Verify the proper PMT and filter(s) are being used for the desired analysis and that the filter is not loose. If loose, add an extra O-ring.
	HV too high	Lower the HV setting.
	Ignitor cap not properly sealed	Secure the ignitor cap.
	Trigger level improperly set	Adjust the trigger level.
	Contaminated combustor	Bake the PFPD overnight at 350 °C and observe if the noise is reduced. If the noise is not reduced, replace the combustor.
PMT voltage stays at 0 V	Severe light leak	The PMT may be exposed to ambient light or the PMT is damaged. Replace the PMT.
Negative baseline	Output signal zero set at an inappropriate level.	Reset the autozero via the Board/Channel Parameters screen in PulseView Monitor.
	High gain in the PMT	Reduce the PMT voltage.
High baseline	High contamination	Examine the emission profile for response due to contamination. Replace the combustor if necessary.
	Improperly set output signal zero	Verify the output signal zero is enabled.
	Improper gas flows	Check that H_2:air ratio is optimized and that the range value is correctly set.
	Improperly set gate values	Check gate values and filter-PMT combination.

Truncated peak tops	Detector response exceeding the PFPD's or data system's signal handling capacity	Reduce the electrometer gain by reducing the PMT voltage (if necessary), and increase the attenuation value (normally set to one).
	Range setting for the signal set too low	Increase the range setting.
	Output signal zero offset set too low	Reset the zero value.
	Analyte concentration too high for the column	Use a split injection to reduce analyte concentration.
Truncated peak bottom	Output signal zero set too high	Reset the output signal zero via PulseView Monitor.
	Insufficient PMT voltage (gain)	Optimize PMT voltage for noise measurements (see Chapter 15, "Noise Sources and Output Signal Optimization" for details).

Low sensitivity	Excess flow through the combustor	Adjust the needle valve to tick-tock operation. Back off 1/4 to 1/2 turn.
	Low PMT voltage	Increase the PMT voltage setting (50 V higher) and monitor the noise. If noise remains the same, continue increasing the PMT voltage until the noise level increases (doubles the initial noise measurement). Rerun the analysis. If the signal response increases proportional to the noise, reset the PMT voltage to the original optimized noise level.
	Incorrectly set gates	Check gate start and stop times to ensure they are set correctly for maximum selectivity, and check threshold (trigger) level for proper sensing of the propagating flame event
	Contaminated combustor	Bake the PFPD overnight at 350 °C and observe if the noise reduces. If the noise is not reduced, replace the combustor.
	Wrong filter	Verify the correct filter is being used with the PMT
	Improper gas flows	Verify the H_2 and Air 1 flow are optimized.
	Improper gate values	Verify the correct mode and gate values are being used. Optimize gate and threshold (trigger) level settings in the Board/Channel Parameters screen in PulseView Monitor.

Severe peak tailing	Unswept dead volumes, sample adsorption on activated surfaces, sample decomposition, or a leaking injector septum	Check PFPD temperature to ensure the sample is not condensing prior to elution into the combustor and that the PFPD temperature does not exceed the recommended maximum temperature for the column.
	Crushed column at the combustor support	Check the column end protruding beyond the combustor support to verify full flame propagation into the combustor. Increase the detector temperature to prevent condensation in the detector body. Verify proper operation of the injector and column installation.
	Normal elemental reaction	Peak tailing is normal in As, Sn, and Sb detection. The tail is reduced for these elements at high PFPD temperatures. At low PFPD temperatures, the tail may become much longer but with a low amplitude.
	Ruined column	Replace the column. If dirty samples are used, add a guard column.
Nonuniform response	PFPD too cold	Increase the PFPD temperature to 300 °C for phosphorus or sulfur-phosphorus pesticides.
Reduced sulfur response under coelution with hydrocarbons	Quenching	Replace the column with one with better separation, inject less sample. Use a 3-mm combustor, and increase Air 1.

Chapter 9 Replacement Parts

This chapter provides a list of parts and support items for the 5383 PFPD 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.

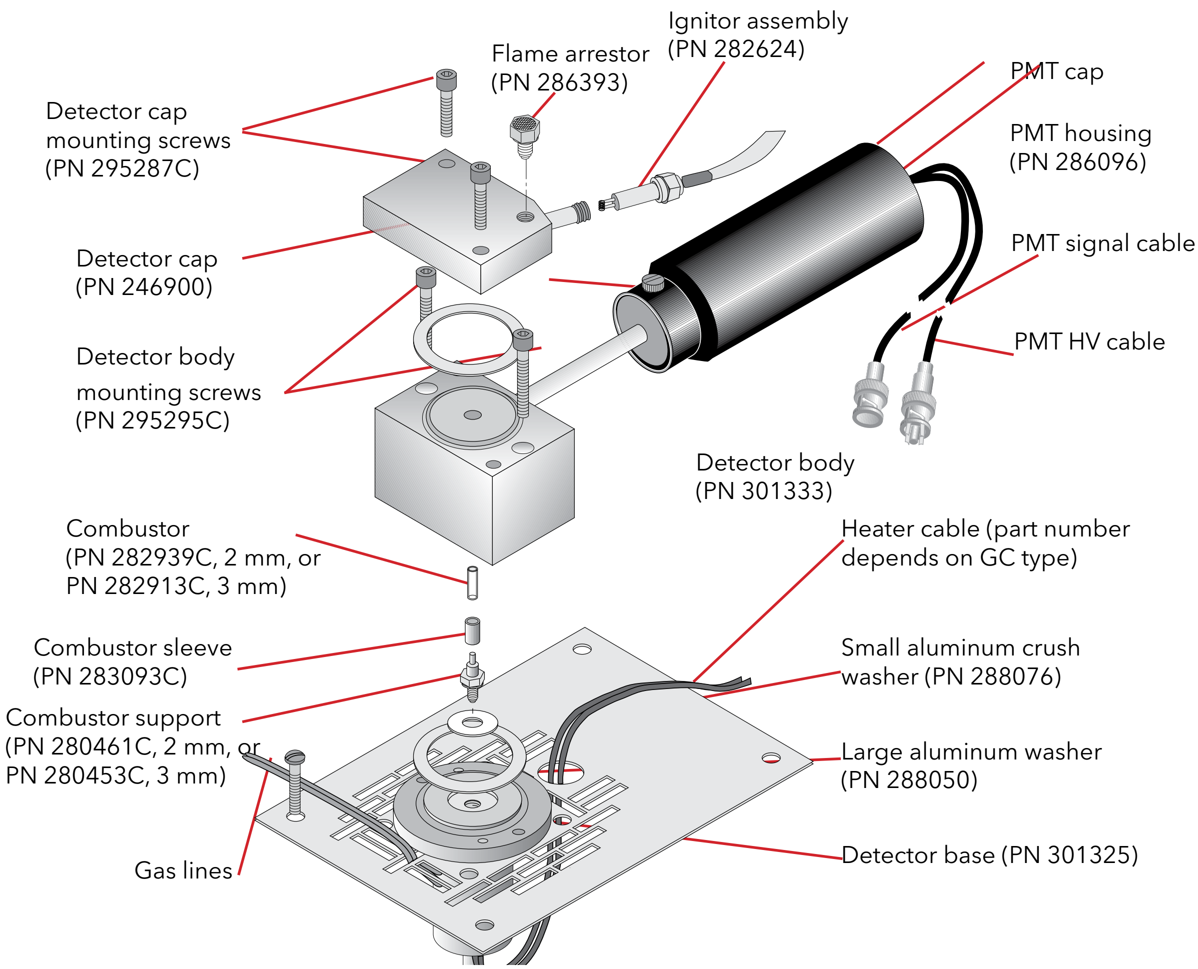


Figure 9.1. PFPD Assembly

Table 9.1. PFPD assembly

Product	Size	Unit	PN	XPN
Barbed adapter fitting	10-32 x 1/16 hose	each	202077	
Combustor (Tested)	2 mm	each	282939C	*
Combustor (Tested)	3 mm	each	282913C	*
Combustor sleeve	–	each	283093C	
Combustor support	2 mm	each	280461C	

Combustor support	3 mm	each	280453C	
Combustor support washer, aluminum	–	10/pk	288076	*
Detector base	–	each	301325	
Detector body	–	each	301333	
Detector cap	–	each	246900	
Heater assembly cable for Agilent 5890	–	each	178954	
Heater/PRT assembly cable for Agilent 6890	–	each	252429	
Heater/PRT assembly cable for Shimadzu 17A	120 V	each	244814	
Heater/PRT assembly cable for Shimadzu 17A	240 V	each	299933	
Heater/PRT assembly cable for Shimadzu GC-2014 or 2010	120 V	each	324388	
Heater/PRT assembly cable for Shimadzu GC-2014 or 2010	240 V	each	324389	
Ignitor assembly cable	–	each	282624	*
Light pipe	–	each	282608C	
Light pipe clamp	–	each	245563C	
Light pipe O-ring, Viton	–	5/pk	288068	*
Lock washer	–	each	132654	
Mounting plate for Agilent 5890	–	each	245464	
Mounting plate for Agilent 6890	–	each	280735	
Mounting plate for Shimadzu 17A	–	each	285916	
Optical filter, BG-12	–	each	282947	
Optical filter, BG-39	–	each	290015	
Optical filter, GG-495	–	each	282921	
Optical filter, RG-695	–	each	290007	
Optical filter, UV34	–	each	310623	
O-ring, large, Viton	–	5/pk	288084	*
Photomultiplier housing assembly	–	each	286096	
Photomultiplier tube, R1924a	–	each	284364	
Photomultiplier tube, R1925	–	each	290049	
Photomultiplier tube, R1925	–	each	290049	
Photomultiplier tube, R5070	–	each	290106	
PMT thumbscrew	6-32 x 1/4"	each	326033	
Screw, detector body to detector base	6-32 x 1"	each	295295C	*
Screw, detector cap to detector body	6-32 x 3/4"	each	295287C	*
Screw, mounting plate to detector base	6-32 x 1/4"	each	295279C	

Screw, stainless steel M4 for mounting to Agilent 5890 and Shimadzu 17A and GC-2014	M4-0.7x8 mm	each	169963	
Screw, Torx for mounting to Agilent 6890 or 7890	–	each	252585	
Temperature sensor assembly cable for Agilent 5890	–	each	170093	
Washer, crush, large, aluminum	–	10/pk	288050	*

Table 9.2. Detector Electronics Controller

Product	Unit	PN
PCF, PFPD 5383 (controller PCA board programmed)	each	330554
Cable, PFPD internal power	each	330557

Table 9.3. Detector Pneumatics Controller

Product	Size	Unit	PN	XPN
Aux EPC pressure control manifold for Agilent 6890	–	each	275974	
Ferrule, stainless steel	1/8" tube	5/pk	210591	
Gas flow controller, air	60 mL	each	282475	
Gas flow controller, hydrogen	25 mL	–	282483	
Gas flow module kit for PFPD on Agilent 6890 Aux EPC	–	each	286344	
Gas flow module kit for PFPD/ OIM for Agilent 6890	–	each	312678	
Gas flow module kit for PFPD on Shimadzu 14 with Aux APC	–	each	324341	
Needle valve, Nupro	–	each	282491	
Nut fitting, stainless steel, male	1/8"	each	112458	
OIM module gas flow controller for Agilent 6890	–	each	285049	
OIM module gas flow controller for Agilent 7890	–	each	324768	
Tee fitting, male	1/16"	each	282509	*

Table 9.4. Miscellaneous Parts

Product	Size	Unit	PN	XPN
Allen wrench	–	–	284539	
Column nut fitting, reverse ferrule type	1/16"	–	223057	*
Combustor extractor tool, 2 mm w/sheath	–	–	280719	*
Combustor extractor tool, 3 mm w/sheath	–	–	280727	*
Dimethyl disulfide permeation tube	–	–	290452	
Dimethyl methyl phosphonate permeation tube	–	–	290460	
External event cable for Agilent 6890 or 7890	–	each	252569	
Ferrule, graphite/Vespel	1/16 x 1/16" I.D. tube	10/pk	216366	*
Ferrule, graphite/Vespel	1/16 x 0.0 mm I.D. tube	10/pk	197079	*
Ferrule, graphite/Vespel	1/16 x 0.4 mm I.D. tube	10/pk	208330	*
Ferrule, graphite/Vespel	1/16 x 0.5 mm I.D. tube	10/pk	196113	*
Ferrule, graphite/Vespel	1/16 x 0.8 mm I.D. tube	10/pk	196105	*
Forceps, Teflon coated	–	each	283051	*
Fuse, 1.25 amp for 115 V or 230 V unit	–	each	298984	*
Handshake cable for Agilent 5890	–	each	185868	
PFPD remote start cable for Shimadzu 17A	–	each	288977	
PFPD remote start cable for Agilent 6890 or 7890	–	each	288902	
Fuse, 1.25 amp for 115 V or 230 V unit	–	each	298984	*
Handshake cable for Agilent 5890	–	each	185868	
Handshake cable for Shimadzu GC-2014 and 2010	–	each	322646	
PFPD remote start cable for Shimadzu 17A	–	each	288977	
PFPD remote start cable for Agilent 6890 or 7890	–	each	288902	
PulseView™ software CD	–	each	330704	
Permeation device kit w/permeation tube for phosphorus	–	each	290429	
Permeation device kit w/permeation tube for sulfur	–	each	290411	

Power Supply-12VDC External Desktop W/DIN	–	each	330709	
Power cord, 110V for North America	–	each	116038	
Cable-USB TO RS422 Converter	5M	each	329241	
Signal output cable to Agilent 5890 AIB	–	each	229641	
Signal output cable to Agilent 6890 AIB	–	each	274852	
Signal output cable to Agilent Integrator	–	each	229633	
Signal output cable to spade lugs	–	each	215962	

Chapter 10 Glossary

The following is a glossary of the most commonly used terms and abbreviations in this manual.

Term	Description
ADC	Analog-to-digital signal converter
Air 1 flow	Air flow that is split between the combustor and wall gas mixture using the fine adjust needle valve.
Air 2 flow	Air flow that is delivered directly to the wall gas mixture.
Alpha	Scaling factor that modifies the gate B integration value when the dual gate technique is employed to obtain PFPD signal output.
Auto zero	Output signal zero value automatically set by PulseView Monitor. This is activated at a specified time during an analytical run.
Channel	Corresponds to a specified PFPD output signal.
Combustor	Transparent quartz tube within which the GC column effluent combusts.
Combustor gas (COMB)	H_2-rich, H_2:air mixture delivered to the combustor.
Combustor sleeve	Metal sleeve that surrounds the combustor base to separate the combustor and wall gases.
Combustor support	Metal fitting that positions the combustor at the correct position for optimum response.
DAC	Digital-to-analog signal converter.
Detectivity	Minimum detectable amount of analyte passing through the PFPD (usually measured in picograms per second). Detectivity is the proper method for evaluating detector performance because it is a measure of the signal-to-noise ratio.
Detector base	Contains the combustor support through which the GC column passes and the heater and temperature sensor, which regulates the detector temperature.
Detector body	Contains the quartz combustor and acts as a support for the light pipe to which the PMT housing assembly is attached.
Detector cap	Contains the ignitor coil and vent through which spent gases are discharged. It provides a support for the ignitor assembly.
Detector controller	Contains the electronics that control PFPD operation and may also contain the pneumatics.
EEPROM	Electronically erasable programmable read-only memory, which stores the PFPD's specified startup operating parameters (specified in PulseView Monitor).
EPC	Electronic pneumatics control module that may be included in the Agilent 6890 or 7890 GC.

Term	Description
Fine adjust needle valve	Controls the proportion of the H_2:Air 1 mixture that flows to the combustor and wall gas pathways.
Gate	Time segment during the flame propagation lifetime used to initiate and stop the integration period of the output signal.
Gate start	Delay from start of flame propagation when integration of signal output is initiated.
Gate stop	Delay from start of flame propagation when integration of signal output is terminated.
GC column	Fused silica tube containing the stationary phase that acts as a solvent for the sample components and through which the sample is passed.
H_2 flow	H_2 flow that is split between the combustor and wall gas mixture using the fine adjust needle valve.
Heater element	Element used to increase the PFPD's temperature.
Ignitor	Heater element used to ignite the flame in the detector cap.
Ignitor current	Electrical current flowing through the ignitor.
Interpolation level	Level at which peak detect for integration starts. Corresponds to value on PulseView Monitor graph.
Light pipe	Quartz rod that transmits chemiluminescence from the combustor to the photomultiplier tube.

Term	Description
Mode	Integrator conditions specified for each signal output channel in PulseView Monitor.
Multiplier	Multiplier for output channels 1 and 2.
Optical filter	Filter used to isolate specific chemiluminescent bands that are able to reach the photomultiplier tube.
Output zero	The zero value set for the output signal. Usually this is some value below the lowest noise signal when the PFPD is set to tick-tock mode.
PFPD	Pulsed flame photometric detector that operates in a periodic fashion generating emission waveforms.
PRT	Platinum resistance thermometer or platinum resistance temperature detector; an RTD (resistance temperature detector) that is made of platinum; also called a Platinum RTD.
Permeation source	Device that releases a constant flow of one or more compounds containing the analyte of interest at a given temperature. This is used to optimize the PFPD operating parameters for the selected analyte.
Photomultiplier tube (PMT)	Converts the chemiluminescent energy resulting from the combustion of the GC effluent into an electrical current.
PMT housing	Contains the optical filter(s), the PMT, and the PMT socket.
PMT HV cable	Delivers the voltage required by the PMT to convert light energy into an electrical current.
PMT signal cable	Delivers the output current generated by the PMT to the printed circuit board contained in the detector controller.
PMT socket	The keyed receptacle for the pins protruding from the back of the PMT.
PMT voltage	Voltage applied to the photocathode of the PMT to convert photon energy to an electrical current.
Pneumatics	The gas lines and gas flow controllers that direct and regulate the flow of hydrogen and air from the gas supply to the detector base.
PulseView Monitor	Software developed to set up and optimize the PFPD operating parameters.
Record	Raw chemiluminescent data between specified start and stop times during the GC's analytical run.
Remote start	Synchronizes PFPD time events with the start of the GC's analytical run.

Term	Description
Selectivity	If the signal response is linear relative to the analyte concentration, detectivity is signal output per unit mass flow rate of the analyte. The PFPD selectivity determines the ratio of the response from one detected element relative to another.
Signal output	The gated integration output sent from the printed circuit board in the detector controller to the data handling device.
Temperature sensor	Embedded in the detector base and allows control of the PFPD temperature.
Tick-tock	Occurs when the propagating flame alternately terminates at the top and bottom of the combustor because the propagating flame reaches the top of the combustor before the combustor gas mixture fills the combustor.
Timed event	An event that is timed to occur during the GC analysis run time.
Trigger level	Corresponds to a specific current being generated by the PMT and is the value at which a pulsed event is first detected.
Viton O-ring	Prevents parts in the PMT housing from moving, and prevents light leakage from the optical elements of the PFPD.
Wall gas (WALL)	Air-rich hydrogen and air mixture delivered to the ignitor.
Zero	The zero value set for the output signal. This is often set at some value below the lowest noise signal when the PFPD is in the tick-tock mode.

Chapter 11 Bibliography

Patent

Amirav, A., Pulsed Flame Detector Method and Apparatus. U.S. Patent 5,153,673, Israel Patent 95617, Europe Patent 0475250, and Japan Patent 2759854.

Pulsed Flame Photometric Detection

Amirav, A. and Jing, H. "Pulsed Flame Photometer Detector for Gas Chromatography." *Anal. Chem*. **1995**, 67, 3305–3318. Describes improved PFPD pneumatics design, theory relating to repetition rate, dual gate techniques, and experimental information for optimizing operating parameters.

Amirav, A. and Jing, H. "Simultaneous PFPD-MS Detection for Enhanced GC-MS Pesticides Analysis Capabilities." *J. Chromatog*. **1998**, 814, 133–150. Describes how the PFPD improves the performance of MS pesticide analysis.

Atar, E.; Cheskis, S.; and Amirav, A. "Pulsed Flame: A Novel Concept for Molecular Detection. *Anal. Chem.* **1991**, 63, 2062-2064. Introductory paper on the PFPD.

Cheskis, S.; Atar, E.; and Amirav, A. "Pulsed Flame Photometer: A Novel Gas Chromatography Detector." *Anal. Chem*. **1993**, 65, 539-555. Describes PFPD principles.

Jacobsen, J.A.; Stuer-Lauridsen, F.; and Pritzl, G. "Organotin Speciation in Environmental Samples by Capillary Gas Chromatography and Pulsed Flame Photometric Detection (PFPD)." *Appl. Organometallic. Chem.* **1997**, 11, 737–741. Describes environmental organotin analysis with the PFPD.

Jing, H. and Amirav, A. "Pesticide Analysis with the PFPD and a Novel Direct Sample Introduction Device." *Anal. Chem*. **1997**, 69, 1426–1435. Describes pesticide analysis with the PFPD and dual gate manipulations.

Jing, H. and Amirav, A. "Pulsed Flame Photometer Detector—A Step Forward Toward Universal Heteroatom Selective Detection." *J. Chromatogr.* **1998**, 805, 177–215. Provides details of selectively detecting 28 elements with the PFPD.

Kalontarov, L.; Jing, H.; Amirav, A.; and Cheskis, S. "Mechanism of Sulfur Emission Quenching in Flame Photometric Detectors." *J. Chromatogr. A*. **1995**, 696, 245. Explains quenching and quenching mechanism for FPDs and PFPDs.

Tzanani, N. and Amirav, A. "Combined Pulsed Flame Photometric Ionization Detector." *Anal. Chem*. **1995**, 67, 167–173.

Other Important References

Zainullin, R.F. and Berezkin, V.G. "Flame Photometric Detectors in Chromatography: A Review." *Critical Reviews in Analytical Chemistry*. **1991**, 22, 183–199.

Farwell, S.O. and Barinaga, C.J. "Sulfur-Selective Detection with the FPD: Current Enigmas, Practical Usage, and Future Directions." *J. Chromatographic Science*. **1986**, 24, 483–494.

Chapter 12 Photomultiplier Tube and Filter Configurations for Various Element Detection

This chapter provides a list of filters that can be installed in the PMT to detect different species produced

Element	Photomultiplier Tub (PMT)	PMT PN	Filter	Filter PN
Sulfur (2 mm)	R1924 (included with PFPD)	284364	BG-12 (purple) (included with the PFPD)	282947
Sulfur	R1925	290049	BG-12 (purple)	282947
	R5070	290106		
Phosphorus (3 mm)	R1924	284364	GG-495 (yellow)	282921
Phosphorus	R1925	290049	GG-495 (yellow)	282921
	R5070	290106	BG-39 (light blue)	290015
Nitrogen	R1925	290049	RG-695 (black IR)	290007
	R5070	290106		
Sulfur and phosphorus (3 mm)	R1924	284364	UV-34 (clear)	310623
Sulfur and phosphorus	R1925	290049	UV-34 (clear)	310623
	R5070	290106	BG-39 (light blue)	290015
Sulfur and nitrogen	R1925	290049	BG-3 (blue)	302919
	R5070	290106		
Phosphorus and nitrogen	R1925	290049	GG-495 (yellow)	282921
	R5070	290106		
Sulfur, phosphorus, and nitrogen	R1925	290049	UV-34 (clear)	310623
	R5070	290106		
Tin (3 mm)	R1924	284364	BG-12 (purple)	282947

Chapter 13 Defining Detectivity

Detectivity (detectability) results from both the peak-to-peak noise level and the sensitivity (response) of the 5383 PFPD to a particular species. The following definitions apply to both flame photometric detectors (FPDs) and pulsed flame photometric detectors (PFPDs) and are based on ASTM Standards E840-95(2000).

Phosphorus Mode

The minimum phosphorus detectability is the mass flow rate of phosphorus atoms in the carrier gas that gives a detectable signal equal to twice the peak-to-peak noise level and is calculated from the measured sensitivity and noise level.

Equation 1: $Dp = 2Np / Sp$

where: Dp = minimum phosphorus detectability [pg P/sec]
Np = peak-to-peak noise level in phosphorus mode [Amp]
Sp = phosphorus sensitivity (response) [Amp × sec/pg P]

A generally linear relationship exists between the PFPD's phosphorus response and the mass flow of phosphorous atoms into the flame. Calculate the PFPD's phosphorus sensitivity (response) as follows:

Equation 2: $Sp = Ai / mP$

where: Sp = phosphorus sensitivity (response) [Amp × sec/pg P]
Ai = integrated peak area [amp × sec]
mP = mass of phosphorus atoms in the sample [pg P]

Substituting equation 1 for Sp in Equation 1 gives the following expression, which can be used to calculate detectivity directly.

Equation 3: $Dp = 2Np \times (mP / Ai)$

Sulfur Mode

The minimum detectability for sulfur is the mass flow rate of sulfur atoms in the carrier gas that gives a detectable signal equal to twice the peak-to-peak noise level and is calculated from the measured sensitivity and noise level. Since the sulfur response frequently obeys a square law, calculate the minimum sulfur detectability as follows.

Equation 4: $Ds = (2Ns / Ss)^{1/2}$

where: Ds = minimum sulfur detectability [pg S/sec]
Ns = peak-to-peak noise level in sulfur mode [Amp]
Ss = sulfur sensitivity (response) [amp/(pg S/s)^2]

A generally nonlinear quadratic relationship exists between the PFPD response and the mass flow rate of sulfur atoms into the flame. Determine the sulfur sensitivity as follows.

Equation 5: $Ss = (Ai / mS) \times (1 / Ms)$

where: SS = sulfur sensitivity (response) [Amp/(pg S/s)^2]
Ai = integrated peak area [Amp × sec]
MS = mass of sulfur atoms in the sample [pg S]
mS = mass flow rate of sulfur atoms in the sample at one-fourth peak height [pg S/sec]

Equation 6: $mS = Ms / W_{1/4}$

where: $W_{1/4}$ = width of the peak at 1/4 peak height [seconds]

Substituting equations 5 and 6 into equation 4 yields an expression for calculating sulfur detectivity.

Equation 7: $Ds = [(2Ns \times M^2s)/(Ai \times W_{1/4})]^{1/2}$

The sulfur response often obeys the square law. Calculate the square root of the response to obtain a linear relationship between sulfur concentration within the analyte and the PFPD signal output. The PFPD's dual channel capability allows simultaneous output of both the direct quadratic sulfur response and the linear square root response signals.

Chapter 14 Dual Gate Capability and Alpha Calculation

OI Analytical's PulseView Monitor and PulseView Analyzer software programs greatly facilitate using the 5383 PFPD's dual gate features. This chapter describes optimizing this advanced technique. If additional information is required, call OI Analytical's Technical Support Department at (800) 336-1911 or (979) 690-1711.

Enhancing Selectivity Using Multiple Gates

For operating simplicity, using a single gate is preferred for determining a combustion product's chemiluminescent response. However, emissions associated with different species may overlap in both the spectral and time domains. This interference limits selectivity for a given analyte. Multiple gate techniques further increase selectivity when the interfering species do not significantly affect the time and spectral characteristics of the analyte or flame propagation. In particular, emissions from analytes with substantially different time domains can be easily separated.

Necessary criteria for successfully using multiple gate techniques are:

- Sufficiently large differences in the emission-time domain of each species allow specifying discrete gates, each of which incorporates a substantial segment of the time domain of one emitting species only,
- Gate conditions are held constant,
- Gas composition, spectral filters, and PMT conditions are held constant.

The 5383 PFPD's signal processor (i.e., controller board) incorporates 250 discrete information channels along the 25-millisecond emission-time profile. The output from each channel depends on the flame composition, which is determined by the relative flow rates of hydrogen, air, carrier gas, and analytes, and the spectral sensitivity of the optical filter and PMT. The basic premise for using multiple gates is that the observed emission in each discrete information channel is the sum of independent emission-time profiles of each emitting species.

Using the Dual Gate Option in PulseView Monitor

To avoid confusion, phosphorus and sulfur are used in the following example to demonstrate how to increase selectivity between two species using PulseView Monitor software's dual gate capability. The gates used to differentiate the phosphorus and sulfur response modes are referred to as gate P and gate S, respectively. The phosphorus and sulfur chemiluminescent time domains and the gate P and gate S settings are shown in Figure 14.1.

NOTE: Gate P and gate S are distinguished by the two colors (blue and green) of the individual markers.

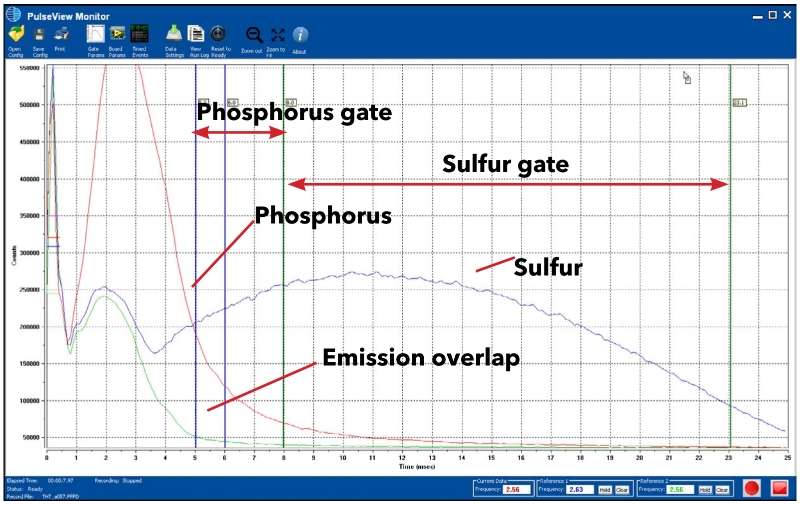


Figure 14.1. Phosphorus and Sulfur Emissions

As seen in Figure 14.2, PulseView Monitor lets the operator specify two gates, A and B, for each mode within the Gate Parameters screen, enhancing PFPD selectivity for two (or more) species with partially overlapping emission-time profiles. To employ this option, gate A must incorporate a substantial segment of the first species' chemiluminescent time domain that does not significantly overlap with the time domain of species two. Conversely, gate B must include a substantial segment of the second species' time domain that does not significantly overlap with the time domain of species one. For example, maximize selectivity for phosphorus when gate A in the phosphorus mode is set to incorporate as much of the phosphorus chemiluminescent time domain as possible while at the same time minimizing the carbon or sulfur chemiluminescent time domain. (In Figure 14.1, the phosphorus gate is set from four to nine milliseconds, which eliminates most of the carbon response and the back end of the sulfur emission.)

To use the dual gate technique to eliminate interference between two species, set reciprocal gate assignments in the PulseView Monitor Gate Parameters screen. Assign gate A to the species of interest and gate B to the interfering species. For example, specify gate A start and stop times for the phosphorus mode to optimize phosphorus response (4–9 ms), while gate A start and stop times for the sulfur mode are set to optimize the sulfur response (9–24.9 ms). Conversely, gate B in each mode specifies the optimal gate settings for the interfering species (i.e., sulfur in the phosphorus mode and phosphorus in the sulfur mode). Reasonable gate A start and stop times for phosphorus and sulfur are shown in Figure 14.2.

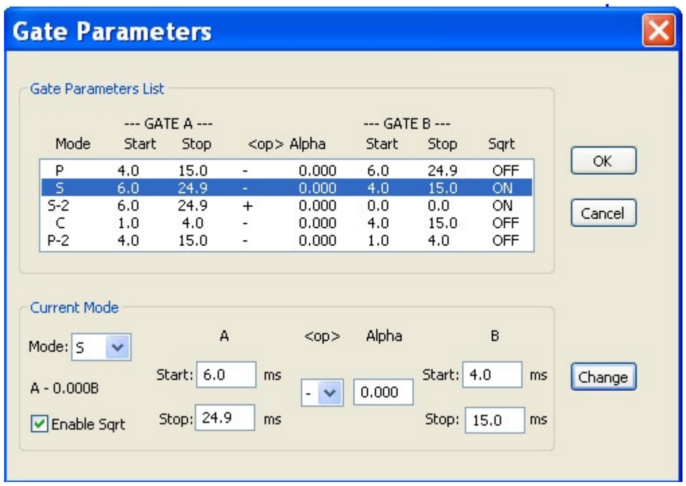


Figure 14.2. Gate Parameters Window Showing Gate Settings for Phosphorus (P) and Sulfur (S)

The PFPD provides two separate analog signal output channels to assign the phosphorus mode to channel 1 and the sulfur mode to channel 2. When setting the Alpha value in each mode to zero (Figure 14.2), channel 1 provides the output in the phosphorus mode (i.e., gate P output), and channel 2 provides the output in the sulfur mode (i.e., gate S output). Thus, the output signals from phosphorus and sulfur in channels 1 and 2 are directly proportional to the concentration of each species in the combusted analyte and the average emission coefficient of each species within gate P and gate S, respectively.

Figure 14.3 shows channels 1 and 2 chromatograms derived using the Figure 14.2 gate parameter values together with a pesticide test standard containing tributyl phosphate (TBP) and tetrahydrothiophene (THT). Because the Alpha values are set to zero in this example, the PFPD's interheteroatom selectivity is not optimized using dual gate enhancements.

The first (left) peak in each chromatogram in Figure 14.3 represents the compound with only sulfur (i.e., THT, which contains no phosphorus). A sulfur peak appears in the phosphorus mode because the phosphorus gate (gate P, which is output in channel 1) contains part of the overlap between the phosphorus and sulfur emission lifetimes (shaded area in Figure 14.1). Similarly, the phosphorus peak appears in the sulfur mode because the sulfur gate (gate S, which is output in channel 2) also partly includes the overlap between the phosphorus and sulfur emission lifetimes.

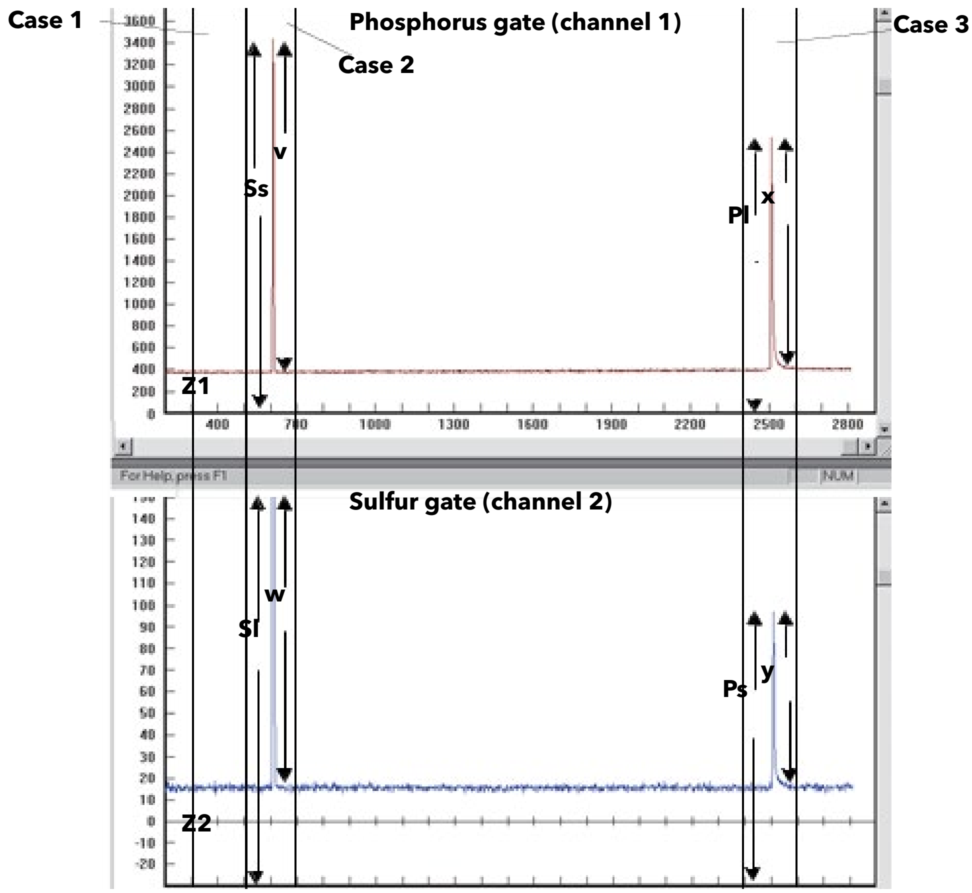


Figure 14.3. Chromatograms from Channel 1 (P gate) and Channel 2 (S gate) Using a TBT/THT Standard

Quantifying Gate Scaling Factors

Determine the optimal value of the gate B scaling factor (Alpha) values for the phosphorus and sulfur modes using either GC iteration (trial and error) as described by Amirav and Jing (see Chapter 10) or empirically using the following procedure:

1. Specify the phosphorus and sulfur mode in the PulseView Monitor Gate Parameters screen shown in Figure 14.2. Set the Alpha value for each mode to zero.

2. Assign the phosphorus mode to channel 1 and the sulfur mode to channel 2 in the PulseView Monitor Board/Channel Parameters screen. Set appropriate zero offsets for both channels (zero offsets do not need to be the same), and set the same attenuation value for each channel.

3. Ensure a suitable filter for detecting the species of interest is installed in the PMT assembly. For example, the chromatogram shown in Figure 14.3 was obtained using the GG 495 filter to enhance selectivity for phosphorus, which was of low concentration relative to sulfur in the TBP/THT test standard. See Chapter 12 for other filters.

4. Configure the GC for a test run to analyze a suitable test standard containing both phosphorus and sulfur. Ensure each compound incorporated in the test standard contains only one of the heteroatoms of interest. Also, heteroatom concentrations in the test standard compounds should be similar to the anticipated concentrations of these species in the substances analyzed. If this is not the case, adjust estimated Alpha values to adequately subtract the chemiluminescence from interfering species. Figure 14.3 shows the chromatograms obtained from the two output channels using a TBP/THT test standard.

5. Conduct a GC analysis of the test standard and obtain a chromatogram for both channel 1 and channel 2 output.

6. Using the two chromatograms, select three segments that represent the following three cases (Figure 14.3).

   - Case 1: No signal peak in either chromatogram. Measure the average signal level of the baseline above zero. The system refers to the average channel 1 signal level as Z1 and the average channel 2 signal level as Z2.

   - Case 2: Small sulfur peak (SS) in channel 1 chromatogram with corresponding large sulfur peak (SL) in channel 2 chromatogram. Measure the heights of the two peaks above zero (Figure 14.3).

   - Case 3: Large phosphorus peak (PL) in channel 1 chromatogram with corresponding small phosphorus peak (PS) in channel 2 chromatogram. Measure the heights of the two peaks above zero (Figure 14.3).

7. Using the values obtained from cases 1, 2, and 3, calculate the Alpha value for the phosphorus and sulfur modes as follows:

   - From cases 1 and 2, calculate the small sulfur peak height above average baseline in channel 1:

     $$v = SS - Z1$$

   - Calculate the large sulfur peak height above average baseline in channel 2:

     $$w = SL - Z2$$

   - To obtain the Alpha value for the phosphorus mode (?P) divide the height of the small sulfur peak by the height of the large sulfur peak:

     $$\alpha p = v/w$$

   - From cases 1 and 3, calculate the large phosphorus peak height above average noise level in channel 1:

     $$x = PL - Z1$$

   - Calculate the small phosphorus peak height above average noise level in channel 2:

     $$y = PS - Z2$$

- Obtain the Alpha value for the sulfur mode (?S) by dividing the height of the small phosphorus peak by the height of the large sulfur peak:

$$\alpha S = y/x$$

The TBP/THT standard generates the two chromatograms shown in Figure 14.3, producing the following results:

v = 24, 159	x = 23.729
w = 65, 350	y = 1, 076
αp = v/w = 0.370	αS =y/x= 0.045

Once these Alpha values are calculated, incorporate them in the phosphorus and sulfur mode specifications in the PulseView Monitor Gate Parameters screen (for example, see Figure 14.4). Specify the operator value (<op> in the Gate Parameters screen) as "–". This signifies that the gate B value is subtracted from the gate A value. The effect of incorporating appropriate Alpha values for each mode is shown in Figure 14.5.

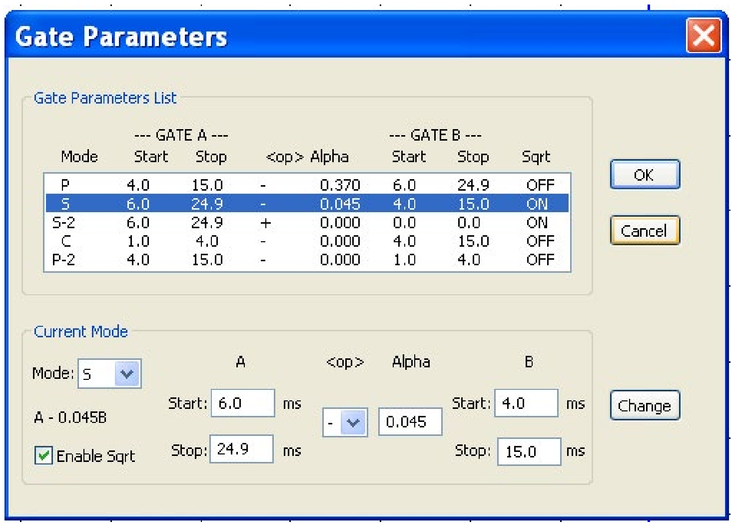


Figure 14.4. Gate Parameter Values for the Phosphorus (P) and Sulfur (S) Mode Including Alpha Values

Comparing the two panels in Figure 14.5 shows that the corrected response of each gate results in the disappearance of the unwanted peak. If the calculated Alpha values result in insufficient compensation to eliminate the unwanted peak or if there is overcompensation (i.e., a negative peak occurs), adjust the Alpha values entered in the Gate Parameters screen to get the desired peak elimination.

OI Analytical's PulseView Analyzer software program, which was used to generate Figure 14.5, greatly facilitates using iteration to derive the best Alpha value. This program allows post-run observation of the chemiluminescent pattern of any point during the chromatographic run, and lets the operator determine how changes to any gate parameter affect the chromatogram.

Providing the filter, PMT voltage, combustor, gas flows, and gate settings are not changed, the estimated chemiluminescence due to the interfering species (i.e., sulfur in the phosphorus mode and phosphorus in the sulfur mode) can now be subtracted from the total channel response. Thus, the channel output response provides an estimate of the chemiluminescent response only of the species of interest (i.e., phosphorus in the phosphorus mode and sulfur in the sulfur mode). This results in a significant enhancement of the PFPD's interheteroatom selectivity.

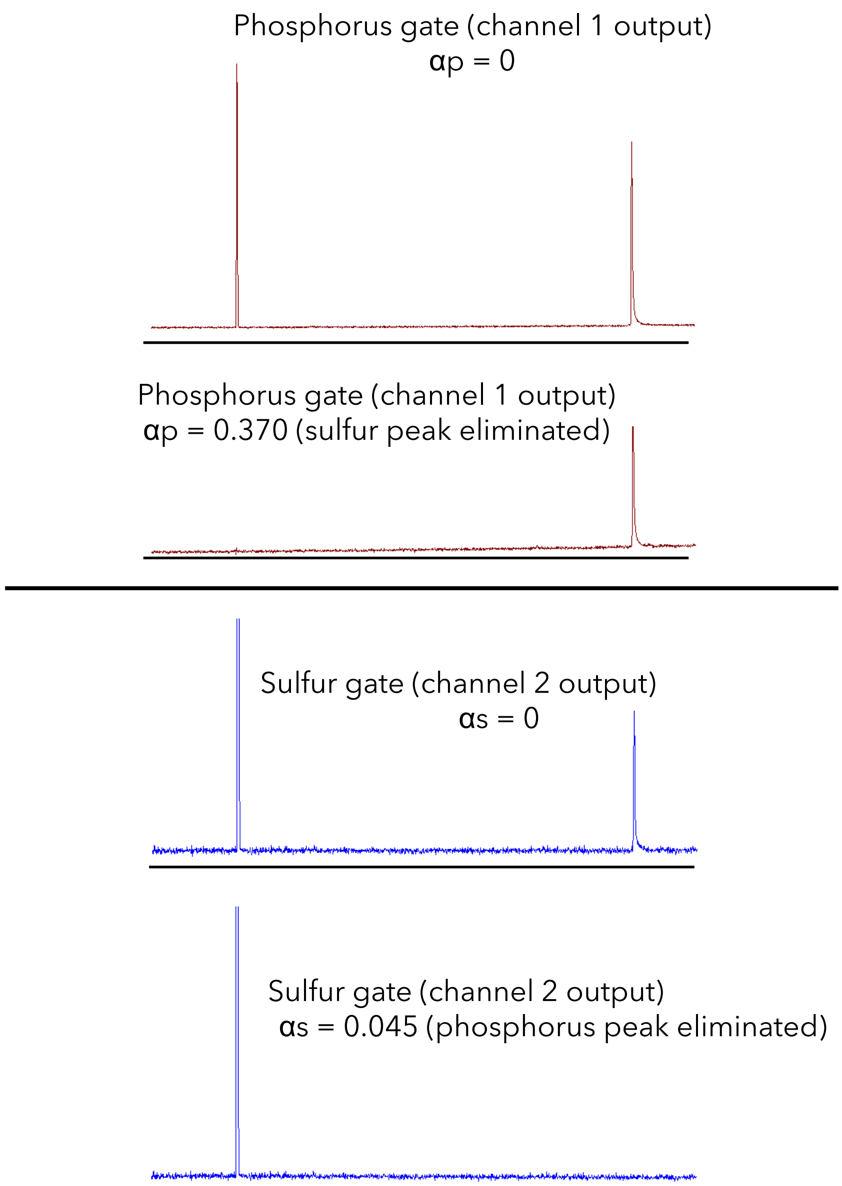


Figure 14.5. Comparing Chromatograms Derived Both With (Upper) and Without (Lower) Using Calculated Alpha Values

Chapter 15 Noise Sources and Output Signal Optimization

This chapter describes noise sources that can affect the PFPD's detectivity and how to optimize PFPD signal output for better performance.

The magnitude of other noise sources within the detector system and the quantitation (resolution) capacity of signal converters determine the extent to which actual output signal noise can be detected. To understand the effects of noise sources on the output signal, a brief summary of the chemiluminescence-to-output data conversion process is useful. The PMT converts photon emissions that reach the light tube to an electrical current according to the PMT voltage. The electrometer within the detector controller converts this current from the PMT to a voltage potential. This voltage signal is amplified according to the range setting. The analog-to-digital converter (ADC) converts this amplified voltage signal to a digital signal. The amplified, digital signal is smoothed using linear or spline interpolation, and the resulting signal is further scaled by the attenuation setting. The digital-to-analog converter (DAC) converts the scaled digital signal to a 0–1 V signal, and the detector controller sends the 0–1 V signal to the data handling device. Increasing the PMT gain (PMT voltage), decreasing the electrometer amps/volt range (range), or decreasing the DAC attenuation (attenuation) amplifies the PFPD output signal, including noise signals.

Noise sources that affect the PFPD's chemiluminescence-to-output data conversion process include PMT dark current, PMT bias voltage stability, electrometer noise, ADC noise, DAC noise, cable noise, and integrator noise. All of these noise sources contribute to the noise background observed on the PFPD, but of greatest concern are the electrometer, ADC, DAC, and background contamination noise sources.

Electrometer Noise

Electrometer noise associated with these current-to-voltage conversions and subsequent amplification is measured either as the root mean square (rms) or peak-to-peak (pp) value of the input current in pico amps.

ADC Noise

The 5383 PFPD digitizes the PMT signal using a 24 bit resolution ADC (0-10 µA f.s. input) with an effective 20 bits of resolution (an ADC count increment of 0.6 pA). Noise associated with the ADC usually is less than 9 pA (rms).

DAC Noise

The DAC (0–1 V output) used in the PFPD also has greater than 20 bits of resolution with a DAC count increment of 60 nV. Generally, noise associated with the DAC has less than 50 counts (rms). Actual system noise may not be observed due to the Chromatographic Data System's ADC's limited input resolution, and associated input noise.

Optimizing Multiplier Settings

To ensure accurate measurement of the output noise, increase the multiplier for the associated channel by factors of two until the apparent noise level doubles, and then set the multiplier back by one factor of two. This action ensures that the observed noise is real and corresponds to actual PFPD noise, and optimizes the dynamic range for the PFPD. Use the multiplier to amplify the output but be aware that the multiplier also decreases the observable dynamic range by the same factor. The optimal multiplier setting is dependent upon the application: utilize the lowest multiplier compatible with the analysis requirements.

Other Factors Contributing to Detector Noise

PulseView Monitor allows examining the gate act in the time base for pulse-to-pulse stability may, in part, be due to marginal ignition and can be eliminated by increasing the ignitor current.

Appendix A: Additional PulseView Analyzer Uses

PulseView Analyzer stores all the pulsed flame emissions during the analytical run. This enables the operator to improve several important PFPD analytical capabilities by manipulating the two gates.

For more information on the dual gate approach for enhanced PFPD operation, read the following paper:

Amirav, Aviv; Jing, Hongwu. Pulsed Flame Photometric Detector for Gas Chromatography. *Anal. Chem.* **1995**, 67, 3305– 3318.

The dual gate approach and post-run data analysis with PulseView Analyzer are used to enhance the PFPD performance.

Optimal Gate Delay for Best Sensitivity or Selectivity

PulseView Analyzer is used for post-acquisition optimization of the gate start and stop set points. These gate settings can have a significant effect on the results. Either the sensitivity or the selectivity, or both, are optimized using PulseView Analyzer.

Simultaneous Sulfur and Carbon Analysis

Simultaneous sulfur and carbon analysis are important for petrochemical analyses. A normal gate (from 6 msec to 24.9 msec) is used for detecting the sulfur, and a second, narrower gate is used for detecting the carbon (from 1.5 msec to 3 msec). If the carbon and sulfur emissions overlap, a delayed start time are set for the sulfur (7 or 8 msec,), this improves the sulfur selectivity without sacrificing sulfur detectivity. However, it will be necessary to use the dual gate subtraction technique to remove the contribution of the sulfur from the carbon emission. The sulfur emission usually exists to a small degree within the carbon gate, and a small alpha coefficient is sufficient to remove the sulfur contribution. PulseView Analyzer is used to determine the best start time for the sulfur gate and the best alpha coefficient for subtraction of the sulfur contribution from the carbon gate.

Simultaneous Multi-element Analysis

Compounds containing both sulfur and phosphorus, such as some pesticides, are analyzed by using a UV-34 optical filter. The dual gate subtraction approach is used to generate simultaneous, single element chromatograms for both heteroatoms. (See Chapter 5 for more information on dual gate subtraction.) Additional optical filter/PMT configurations are available for simultaneous analysis of other heteroatom pairs, such as sulfur and nitrogen or phosphorus and nitrogen.

Reducing Sulfur Interference in Phosphorus Pesticide Analysis

The emission times of specific heteroatoms often overlap (e.g., the tail end of the phosphorus (P) emission overlaps with the initial phase of the delayed sulfur (S) emission). The selectivity for the delayed, extended S emission relative to the earlier P emission are enhanced by simply moving the start S gate to a point beyond the interfering P emission (e.g., 16–20 msec). This improvement in the selectivity between phosphorus and sulfur will result in a small decrease in the sulfur sensitivity. Eliminating the S emission from the P emission with a simple adjustment of gate settings is not as easy because the S emission overlaps with a significant portion of the phosphorus emission.

However, using the dual gate enhanced mode of operation allows the operator to significantly increase phosphorus selectivity relative to sulfur (and vice versa).

This optimization of gates is easily done using the PulseView Analyzer software after running just a single analysis. Once optimal dual gate parameters are obtained, they are simply entered into the gate table of the PulseView Monitor program and used for all subsequent analyses. Application Note #1127 gives a detailed description of how to enhance selectivity for phosphorous versus sulfur or carbon.

Elemental Identification with the Gated Response Ratio (GRR)

The GRR technique is a qualitative technique that is used to identify whether there are multiple heteroatoms present within a specific chromatographic peak without going through the full dual gate subtraction procedure. The GRR technique is particularly useful where complex sample matrices make individual peak identification difficult (e.g., identification of organophosphorous pesticide residues in food matrices containing sulfur). To determine the GRR, a single run is made with compounds containing known concentrations of the heteroatoms of interest. The ratio of the peak heights is a unique number that can then be used to identify whether one or both of the heteroatoms are present. To use the GRR technique for an unknown, one gate is defined for the compound of interest (e.g., 4 msec to 10 msec for P), and a second gate is defined as the P/S ratio (see Figure A1). Comparing the P chromatogram to the P/S chromatogram will then provide information about whether there is sulfur interference present in the target phosphorus peak.

Generally, sulfur and phosphorus are identified by their emission even without any GRR; however, at low levels or if both elements are in a given compound, the GRR may be the best way to confirm this information. The process is qualitative only; it will only indicate whether one or two specific heteroatoms are present in a given compound peak (e.g., S, P, or S and P). The GRR technique will not give quantitative information about the relative amounts of the individual heteroatoms; for quantitative results the dual gate subtraction technique is necessary.

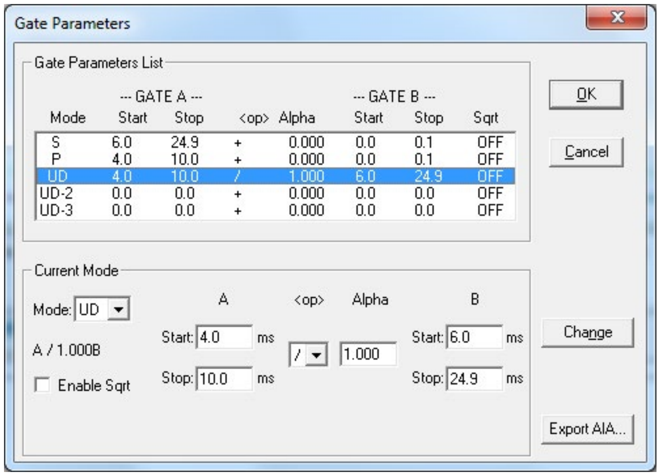


Figure A1. Gate Parameters for GRR Technique for Unknown

Extended Sulfur Measurement Dynamic Range Using the Gated Response Ratio

Due to its quadratic response, the dynamic range for sulfur is limited to about 2.7 orders of magnitude. The sulfur, dynamic range and other elements are extended by approximately one order of magnitude using the GRR. When the sulfur emission becomes saturated, it is easily observed that only a portion of the full sulfur gate has reached a maximum; the early portion of the sulfur emission will not reach maximum or electronic saturation conditions. To use the GRR technique, one gate is defined in the usual manner (e.g., 6 msec to 24.9 msec for sulfur), and a second gate is defined with minimal or no delay and a 2–3msec gate width (see Figure A2). Using a sulfur standard that does not reach saturation conditions, simultaneous chromatograms for both gates are acquired, and the GRR is calculated by dividing the response from the first (normal) gate by the response from the second (early) gate. Then, when a normal sulfur signal (6 msec to 24.9 msec) is acquired under saturation conditions, the response from the early gate (4–6 msec) is multiplied by the GRR to determine the estimated delayed emission response, thus extending the overall dynamic range.

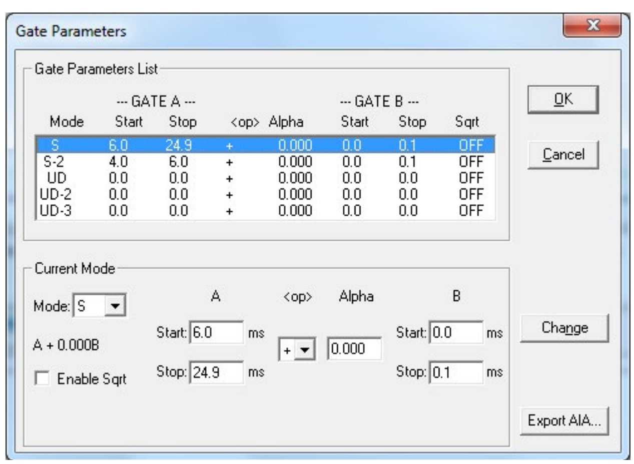


Figure A2. Gate Parameters for Extended Sulfur Measurement

Quenching Identification and Elimination

Hydrocarbon quenching occurs when a high concentration of hydrocarbon co-elutes with the targeted sulfur compound and causes a reduction in or loss of the sulfur emission due to formation of carbonyl sulfide.

Adjusting the chromatographic conditions so that the hydrocarbon and the sulfur peak of interest are separated spatially can eliminate hydrocarbon quenching. It is reduced or eliminated by using a 3-mm I.D. combustor and an increased Air 1 flow rate. Using the larger combustor increases the combustion volume, and the increased Air 1 flow rate reduces the potential for formation of carbon monoxide, and thereby reduces the subsequent reduction in sulfur emissions due to carbonyl sulfide being produced.

PulseView Analzyer is used directly to observe the emission profile for any area of the chromatogram where quenching is suspected. If the characteristic sulfur emission collapses or folds in on itself (i.e., does not extend beyond 20 msec) and is immediately and simultaneously replaced by a saturating hydrocarbon emission, hydrocarbon quenching has taken place.

The GRR technique can also be used to determine qualitatively whether quenching has occurred due to self absorption of the emission by excited state sulfur dimers by ground state sulfur dimers.

The term self-quenching is used to describe the quenching effect seen when there are very high concentrations of sulfur present. In this case, the concentration of sulfur dimers in the combustor is high enough that the ground state sulfur dimers absorb or quench the emissions before they can reach the light tube, and be transmitted to the PMT. When this type of quenching occurs, the top of the chromatographic peak will be shaped like the letter M, having the appearance of a split peak. This type of quenching is easily identified using PulseView Analzyer by observing the change in the characteristic sulfur emission for a suspected peak. The sulfur emission will reach saturation and then collapse, but it will not be replaced with a simultaneous hydrocarbon emission as described earlier. Using a 3-mm I.D. combustor also serves to reduce the chances of self-quenching.

The two gates are defined, one for the normal sulfur emission (6 msec to 24.9 msec) and one for the early segment of the emission (6 msec to 9 msec). The GRR for the two responses is calculated for a non-quenching (and non-saturating) peak. Comparing the peak heights from the two chromatograms acquired for an unknown can easily identify quenching. The GRR will be changed by quenching, and this change will be the indication that quenching has occurred.

Appendix B: Optimization of the PFPD Offset

The PFPD Square Root Function

The 5383 PFPD has a square root function, which can be used to help linearize the detector response for modes that produce a quadratic response in the detector (S, Br, Se, Te). This function operates by taking the square root of the detector response obtained using the operating parameters setup for the detector.

It should be noted however that due to the PFPD's waveform signal processing in the 5383 Controller, and the mathematical limitations on implementing this function, there are certain limitations as to how low a concentration level can be reached and still provide a typical linear calibration curve. The basic functionality that leads to this limitation is related to the **offset** or **zero** value the user sets up in the PFPD controller (using the PulseView software). This zero value (plus a small offset value added into the number to avoid the possibility of taking square roots of negative numbers) has a dramatic influence in a resulting observed flattening of the curve as the response approaches low numbers.

Offset Effects

The effect of an offset being set improperly is illustrated by considering the effect on a quadratic function - $y = a x 2 + b$. The resulting curves are shown in Figures B1 and B2, when the offset or zero is set too large or too small.

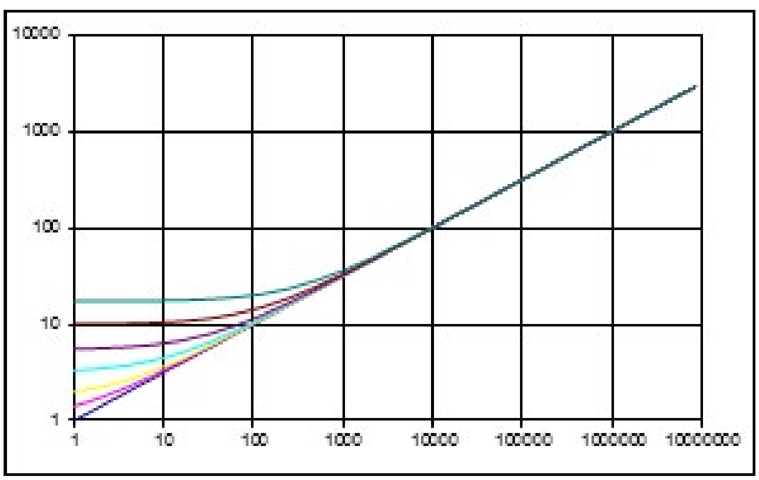


Figure B1. Offset Set to Increasingly Too Small Values

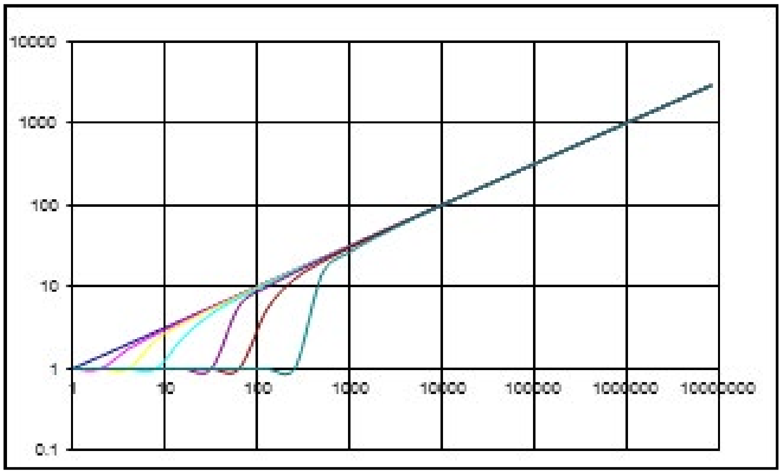


Figure B2. Offset Set to Increasingly Too Large Values

To maximize the range covered detector to a proper value by a linearized response in the PFPD, it is critical to properly set the zero of the detector. The more accurately this is set, the greater the linear range will be. *Regardless of the zero setting, the user should be aware that at the low end of the calibration or response curve the curve will still flatten out due to measurements at or near the noise limit (integration of noise and issues with baseline drift relative to the user set zero offset value)*.

Setting the Offset

Once a set of gate parameters have been set, the PMT voltage and trigger level have been set, and the gas flows have been optimized, perform the following procedure using the PulseView software in conjunction with the chromatographic acquisition program (e.g., Chemstation) to optimize the maximum range for the linear response on the PFPD. The simple objectives are to determine the minimum multiplier setting (to ensure that PFPD output is not limited by the analog to digital resolution of the chromatographic data system), and to set the output to zero so that the observed noise is above the zero setting for the chromatographic data system.

Allow the system to warm up (i.e., thermally equilibrate) and the system to stabilize for a minimum of 10 minutes (after start up) before performing the following steps.

1. Monitor the correct output channel. Verify that the square root function is turned **on** in the gate parameters windows for the desired mode, and that the mode is selected in the board parameters window.

2. Verify that there is no sulfur output being observed in the detector waveform. This is done by zooming the PulseView graphics display to monitor the detector output so as to observe the noise in emission profile. By putting the detector into tick-tock mode (see Chapter 6) one can readily observe whether there is any background sulfur in the GC system or detector. This is an important step because any background S response will also result in a non-linear response due to this additional S background contributing to the detector signal.

3. In the Board Parameters screen in the PulseView software, set the zero offset for the appropriate channel to zero, and apply the change. Click the **About** button to open a window that updates the individual channel results on a pulse by pulse basis. Verify that a non-zero value is being displayed for the specific output channel. Monitor the output values for 30-50 seconds and keep track of the 'minimum value'. Verify this on the chromatographic integrator (workstation), and that 'noise' is being clearly observed (i.e., the detector signal is greater than 0 on the display or as noted in the detector baseline in the data handling system). Adjust the integrator/workstation as necessary to visually observe the detector output.

4. Set the multiplier for the output channel to 1. Monitor the baseline for 1 to 2 minutes. Next, increase the multiplier a factor of two (for the same channel), and again monitor the output for 1 to 2 minutes. If the peak-to-peak noise of the signal does not double, continue increasing the multiplier a factor of two until it does. The multiplier that increased the peak-to-peak noise a factor of two is the recommended multiplier value. This step ensures that the PFPD output is not limited by the resolution of the chromatographic data system.

5. Set the zero value to a value much greater than that being displayed per the channel output in the PulseView About window. If the display is showing values ranging from 70 to 300, set the value to 600. The displayed value in PulseView should now be showing zero, and the chromatographic output should shift to a lower output signal level, with less noise. This results from forcing the output to a zero output level. Steps 3, 4, and 5 together show the bounds of the system (for a zero offset with no sulfur response). The objective now is to set the zero (for the channel of interest) to a value where the noise sits just on the level observed in step 4.

6. Iteratively set the zero value (in board channel parameter) for the channel of interest to the lowest displayed value for the channel output, and again verify that noise is being clearly chromatographically displayed. If the noise is being truncated at the bottom, i.e., the noise is flatlining on the bottom of the chromatographic trace, use the set zero function and decrease the observed value until the entire noise is just being displayed. Otherwise, use the set zero function and increase the zero offset value until the noise is just being truncated or sits just above the zero baseline observed in step 5 (repeat step 5 to re-establish the zero baseline if necessary for reference). This zero offset value is the optimum offset for these gate parameters, and chromatographic conditions.

NOTE: *If the chromatographic conditions change, the gate parameters are changed, gas settings, filters, or PMT voltage is selected, the optimum offset will most likely also change and would need to be reset as noted above*. Trade-offs are usually required due to temperature programming of the column, resulting in the pulse waveform changing due to a change in flame stoichiometry in the combustion tube. These effects can be compensated for by decreasing the zero offset value 5 to 20 (actual value depends on PFPD parameter settings) units below the optimum value.

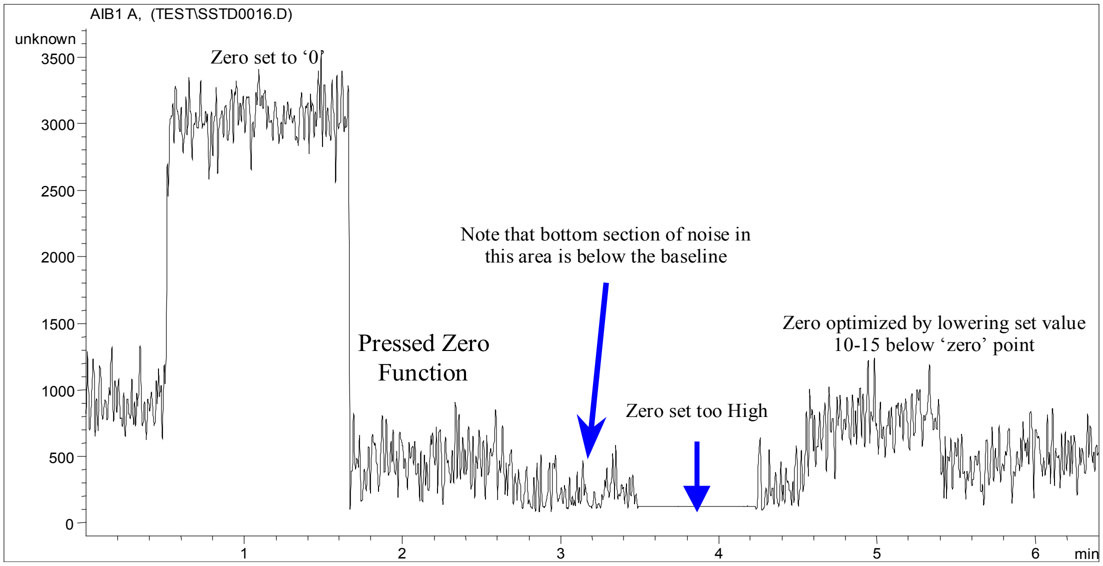


Figure B3. Proper Setting of the Zero Offset

By following these procedures one should obtain calibration curves as noted in Figures B4-5 below.

NOTE: These curves were obtained using a 3-mm combustor versus the 2-mm, which is generally used to maximize S sensitivity. The 3-mm combustor with a slight increase in the amount of air in the air/hydrogen ratio will also help to minimize quenching.

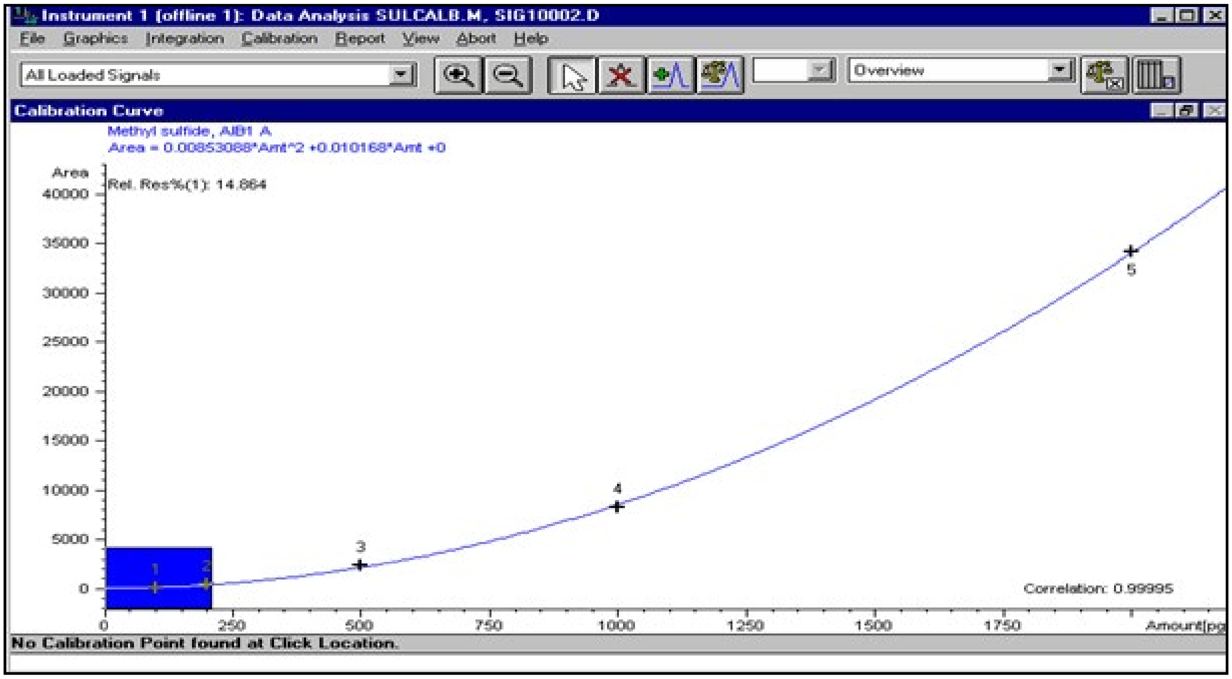


Figure B4. Square Root ON Calibration Curve – Linear Fit

NOTE: The curve will still exhibit some quadratic curvature.

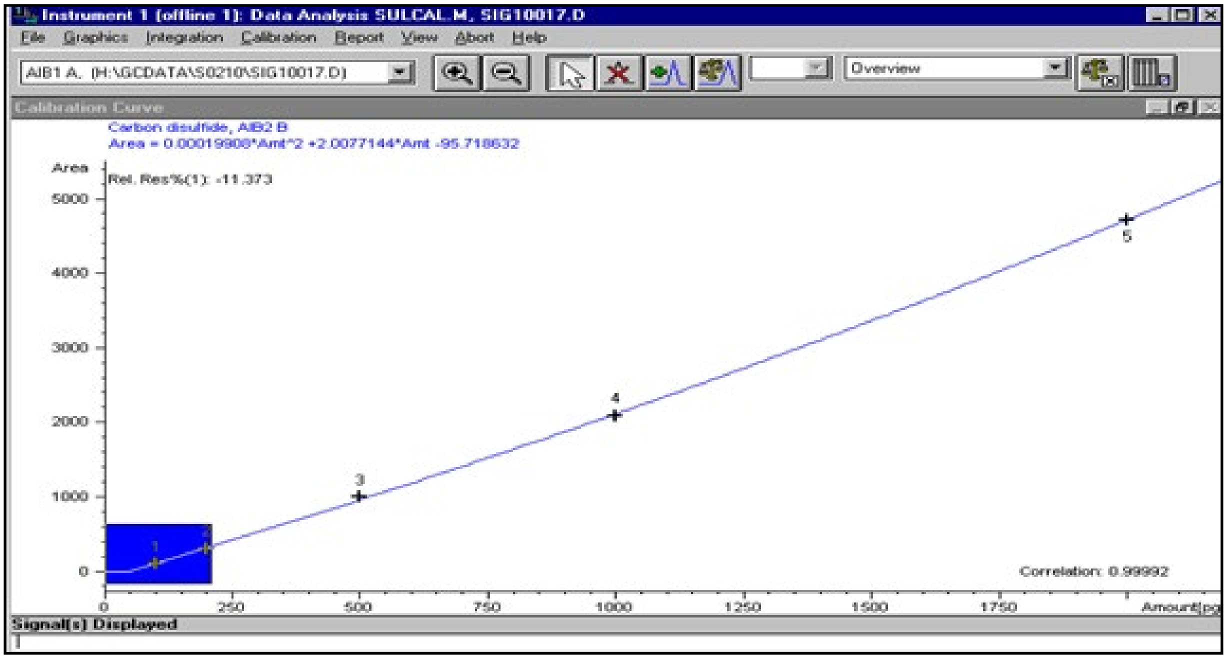


Figure B5. Square Root OFF Calibration Curve – Quadratic Fit

Figure 2.1. Eclipse Front Exterior Components

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Lab Solutions

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