

# **Agilent G2333B (Single) and G2334B (Dual) Flame Photometric Detectors**

**For Agilent 6890 GC**

**Installation and Operation**



**Agilent Technologies**

# Notices

© Agilent Technologies, Inc. 2004, 2005

No part of this manual may be reproduced in any form or by any means (including electronic storage and retrieval or translation into a foreign language) without prior agreement and written consent from Agilent Technologies, Inc. as governed by United States and international copyright laws.

## Manual Part Number

G1535-90328

## Edition

Fourth edition, May 2005

Third edition, April 2005

Second edition, August 2001

First edition, May 2000

Printed in USA

Agilent Technologies, Inc.

2850 Centerville Road

Wilmington, DE 19808-1610 USA

## Acknowledgements

Torx® is a U.S. registered trademark of Textron, Inc.

## Warranty

The material contained in this document is provided "as is," and is subject to being changed, without notice, in future editions. Further, to the maximum extent permitted by applicable law, Agilent disclaims all warranties, either express or implied, with regard to this manual and any information contained herein, including but not limited to the implied warranties of merchantability and fitness for a particular purpose. Agilent shall not be liable for errors or for incidental or consequential damages in connection with the furnishing, use, or performance of this document or of any information contained herein. Should Agilent and the user have a separate written agreement with warranty terms covering the material in this document that conflict with these terms, the warranty terms in the separate agreement shall control.

## Safety Notices

### CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

### WARNING

A **WARNING** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a **WARNING** notice until the indicated conditions are fully understood and met.

# Contents

## 1 Installation

Kit Contents	9
Required Tools	11
Introduction	12
Single-wavelength FPD	12
Dual-wavelength FPD	12
Detector construction	12
Typical Installation	15
Overview	16
Prepare the GC	17
Remove the Covers and Panels	18
Prepare the Detector Location	19
Install the Flow Module	21
Install the Detector	24
Detector body	24
Heater/Sensor cables	25
Signal boards	28
Signal board cable connections	29
Update the Firmware	31
6890A and 6890 Plus	31
6890N	32
Replace the Covers and Panels	33
Return the GC to Operating Condition	34

## **2 Checkout Procedure**

Overview	36
Materials needed	36
Creating Methods	38
Phosphorus method	38
Sulfur method	40
Using ChemStation macros	41
Running the Tests	42
Interpreting Results	46
For phosphorus	46
For sulfur	46

## **3 Operation**

General Rules	48
Starting up	48
Shutting down	48
Photomultiplier (PMT) protection	48
Lit Offset	48
High-purity gases	48
Installing Columns	50
Capillary columns	50
Packed columns, 1/8-inch	51
Packed columns, 1/4-inch	52

## **4 Maintenance and Troubleshooting**

Changing the Wavelength Filter	54
Accessing Heaters and Ignitor	58
Replacing the Heater/Sensor Assemblies	62
Replacing the Ignitor	69

Replacing the Inert Transfer Line	73
Removing the transfer line	73
Reassembly	75
Troubleshooting	76
Offset or detector output too high or too low	76
MDL too high	76
High noise level	77
Small peak area	77
Large peak width at half-height	78
Detector Temperature Not Ready (6890)	78
Clipped peaks	79
Data lost because detector tries to reignite during a run	80
Flame extinguishes after ignition	80

## A Replacement Parts

Inert Transfer Line	84
FPD Ignitor and Heat Shield Assembly	86
FPD Lens Assembly	88
PMT and Bracket Assemblies	90
Covers, Flow Modules, and Electronics	92



# 1

## Installation

Kit Contents	9
Required Tools	11
Introduction	12
Single-wavelength FPD	12
Dual-wavelength FPD	12
Detector construction	12
Typical Installation	15
Overview	16
Prepare the GC	17
Remove the Covers and Panels	18
Prepare the Detector Location	19
Install the Flow Module	21
Install the Detector	24
Update the Firmware	31
Replace the Covers and Panels	33
Return the GC to Operating Condition	34



## **1 Installation**

The G2333B and G2334B kits each contain a flame photometric detector (FPD) for the Agilent 6890 Series gas chromatograph (GC). Table 1 describes each kit further.

**Table 1** FPD installation kits

<b>Kit</b>	<b>Description</b>
G2333B	Contains a single-wavelength FPD, which requires you to change the wavelength filter when switching between sulfur and phosphorus detection modes.
G2334B	Contains a dual-wavelength FPD, which enables you to collect both sulfur and phosphorus data at the same time.

## Kit Contents

Table 2 lists the parts for both kits.

**Table 2** Kit contents

Description	Kit 2333B Single-wavelength	Kit 2334B Dual-wavelength
Auxiliary heater assembly, consisting of:		
• Aux zone/valve box cable	1	1
• Auxiliary heater bracket	1	1
• Screw, M4 × 12 mm, unthreaded shank	2	2
Electronics cover, consisting of:		
• Electronics pan cover	1	1
• Electronics top cover	1	1
• Ground strap assembly	1	1
FPD assembly, (single detector + flow module + board)	1	
FPD assembly, (dual detector + flow module + board)		1
Screw, M4 × 12 mm, threaded shank	5	5
Cable tie	1	1
Hex nut, M4	1	1
Hex nut, 5/16-inch	3	3
ROM set Revision A.03.08	1	1
HOT label warning	1	1
Detector top cover	1	1
Back top panel	1	1
Shipping kit, containing:		
• Silicone tubing, 30-inch	1	1

## 1 Installation

**Table 2** Kit contents (continued)

Description	Kit 2333B Single-wavelength	Kit 2334B Dual-wavelength
• Phosphorus filter	1	1
• Column nut	1	1
• Column tool	1	1
• Ferrule, 1/8-inch, 15% graphite, 85% Vespel	1	1
• Ferrule, No-hole	1	1
• Capillary adapter seat	1	1
• Capillary adapter nut	1	1
• Nut, 1/8-inch, stainless steel	1	1
• Packed column adapter	1	1
• Wrench, 3/8-inch × 7 mm	1	1

### CAUTION

It is neither necessary nor advisable to separate detectors from their flow modules. Doing so can create leaks. Although handling the detector and flow module as a unit is awkward, it can be managed.

## Required Tools

- T-20 Torx® screwdriver
- Knife
- Chip removal tool (Amp 821903-1 or equivalent)
- Column measuring tool (for capillary columns)
- Isooctane
- Column cutter
- Grounded wrist strap
- Clean, lint-free gloves
- Cotton swabs on wooden sticks
- Lint-free cloth
- Diagonal cutters
- 1/4-inch open-end wrench

## **Introduction**

The column effluent is burned in a hydrogen-rich flame. The excited species decay in the cooler region above the flame and emit light. This is viewed by a factory-calibrated photomultiplier tube (PMT). A shield blocks light from the flame itself, while filters isolate the light from sulfur or phosphorus species.

### **Single-wavelength FPD**

You can install one or two single-wavelength FPDs. If you install only one, place it in the back location.

Each single-wavelength FPD has a signal board that mounts in a slot on the right side of the GC. The board for the front detector must be in the front slot; the board for the back detector must be in the back slot.

The signal, high voltage, and ignitor cables from the detector connect to its signal board. The heater/sensor cables (two per detector; one short and one long) plug into connectors on the right side of the GC. Details appear later in this document.

The flow module mounts in the pneumatics chassis at the back-top part of the GC. There are specific module locations for the front and back detector locations, which appear later in this document.

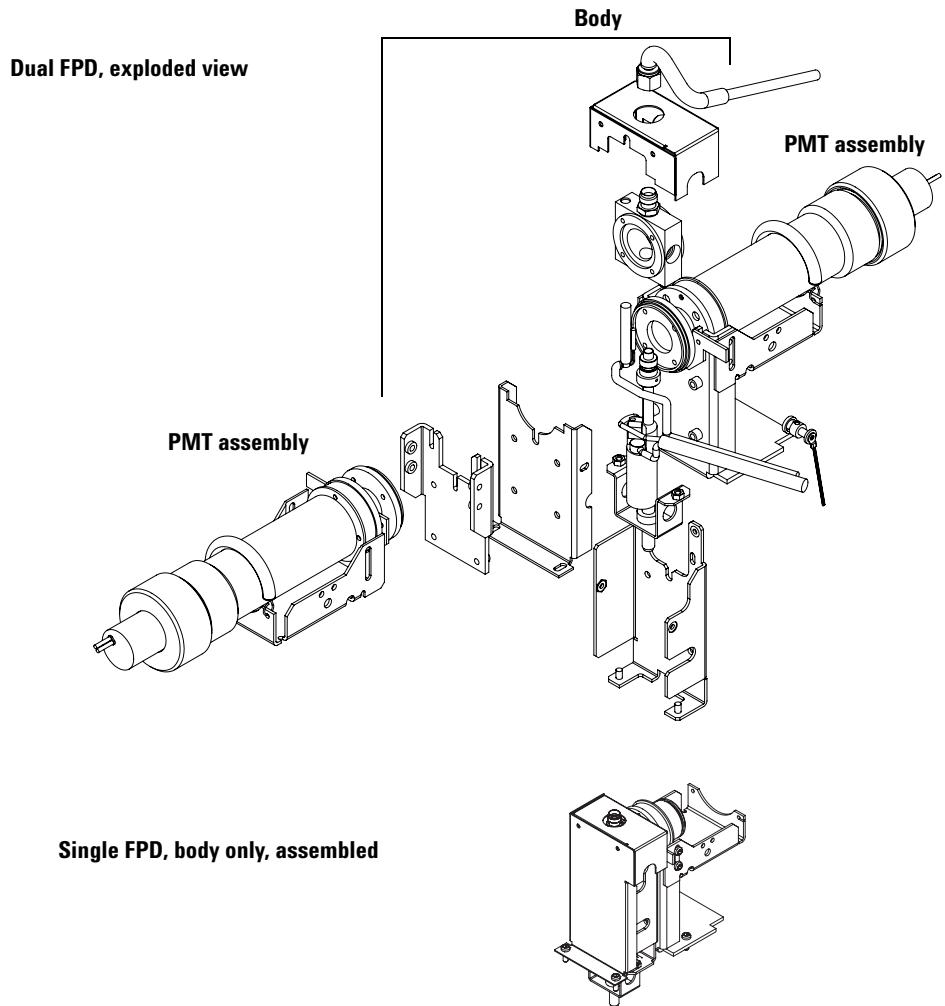
### **Dual-wavelength FPD**

A dual-wavelength FPD has one emissions block assembly and two PMT assemblies. It must be mounted in the back location. It uses two signal boards, one in each slot. The ignitor cable must connect to the board in the back slot. It does not matter which board serves which PMT, but the high voltage and signal cables for a given PMT must go to the same board. The heater/sensor cables go to the positions for a back detector. Details appear later in this document.

The flow module goes in the back detector position.

### **Detector construction**

The FPD consists of a detector body, either single or dual, and one or two PMT assemblies. See Figure 1.



**Figure 1** Single and dual FPD

Most of the illustrations in this document show the single-wavelength body. Parts are identified in Appendix A.

### Detector body

The detector body consists of:

- A base that mounts to the GC
- A transfer line that conducts the column effluent to the emissions block assembly
- A sheet metal chimney that encloses the transfer line
- An emissions block assembly with one (single-wavelength) or two (dual-wavelength) windows and one or two support brackets for PMT assemblies
- One or two lens and filter holders that mount over the emission block windows and connect to the PMT assemblies
- A vent tube to remove gases from the detector and prevent room light from entering the emissions block assembly

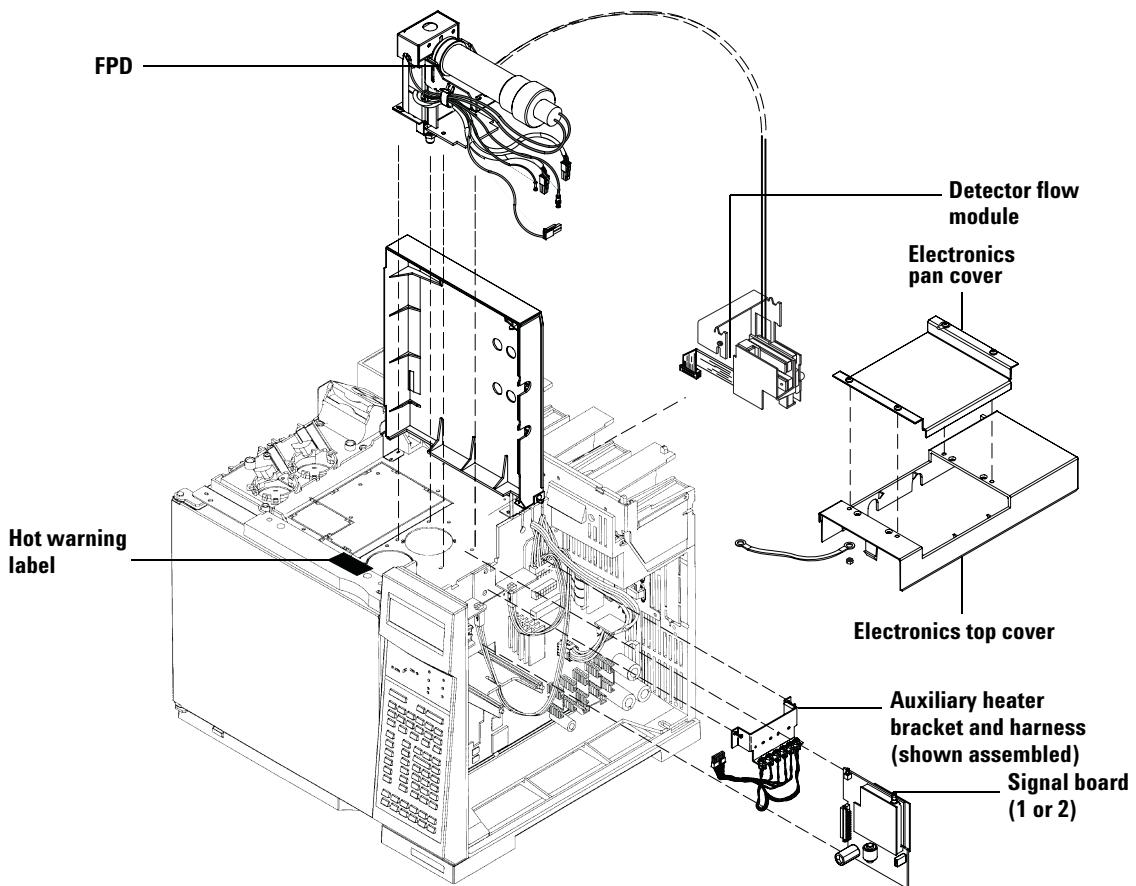
### PMT assembly

A PMT assembly consists of:

- A long tube to place the temperature-sensitive photomultiplier tube well away from the heated emissions block assembly
- An end cap that contains the photomultiplier tube and socket

## Typical Installation

Figure 2 shows the parts for a single-wavelength FPD mounted in the back location. For the front location, the flow module goes in the slot to the right (when facing the back of the GC) of the one shown and the detector and board are moved to the front location and slot.



**Figure 2** A typical single-wavelength installation

A dual FPD will have two of the long photomultiplier assemblies and mount in the back location. There will be two signal boards.

## Overview

**CAUTION**

Perform this installation only if you are experienced in GC maintenance. If not, trained Agilent personnel can install the FPD.

---

- 1** Check the contents of the kit with those listed on page 9.
- 2** Prepare the GC.
- 3** Remove the covers and panels.
- 4** Prepare the detector location.
- 5** Install the flow module.
- 6** Install the detector.
- 7** Update the firmware, if applicable.
- 8** Replace the covers and panels.
- 9** Change the wavelength filter, if necessary.
- 10** Return the GC to operating condition.
- 11** Test the FPD (see Chapter 2, “Checkout Procedure” for more information).

## Prepare the GC

- 1 Turn off the GC and unplug the power cord.

**WARNING**

**Hazardous voltages are present in the instrument whenever the power cord is connected. Avoid a potentially dangerous shock hazard by disconnecting the power cord before working on the instrument.**

---

**WARNING**

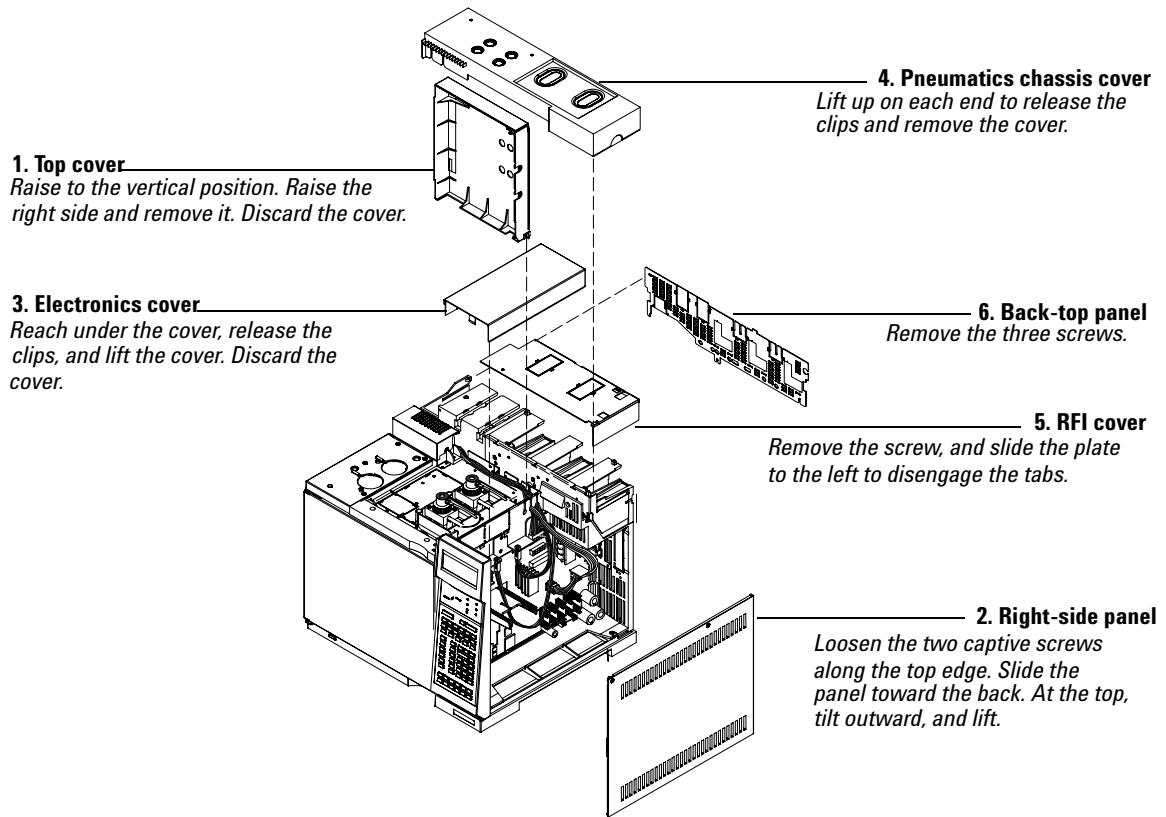
**Hydrogen gas is flammable and potentially explosive. Before installing the detector, turn off the hydrogen gas at the source.**

---

- 2 Turn off all gas supplies.
- 3 Remove columns and hardware associated with both detectors from the oven to clear the way for the FPD.

### Remove the Covers and Panels

You must remove several covers of the GC. Figure 3 shows a suggested order of removal.



**Figure 3** Removing covers and panels of the 6890 GC

## Prepare the Detector Location

### CAUTION

It is neither necessary nor advisable to separate detectors from their flow modules. Doing so can create leaks. Although handling the detector and flow module as a unit is awkward, it can be managed.

- 1 Select the location—front or back—where you will install the FPD. If possible, install it in the back position.

### WARNING

**To avoid damaging the printed circuit board electronics, use a grounded wrist strap (part number 9300-1408) and connect it to a bare metal surface of the GC.**

- 2 If a detector is presently mounted in the selected location, remove it and its flow module. Flow modules are held by a single screw from the front. Remove the screw and slide the module, the detector, and other attachments out the back of the chassis. See the GC user information for details.

If the current detector is to be installed in the other detector location, see that detector's documentation for details.

- 3 Install the hot warning label above the oven door. See Figure 2 on page 15. Align it with the left edge of the large hole and the back edge of the blue bar.

### WARNING

**The insulation on the GC is made of refractory ceramic fibers. Ventilate your work area. Wear long sleeves, gloves, safety glasses, and a disposable dust/mist respirator. Dispose of insulation in a sealed plastic bag.**

### If the detector location has not been used before

- 1 The detector location may be covered by a metal knockout plate. Either cut the webs that hold it or use a screwdriver in the center slot to bend it back and forth until the webs break. Discard the plate.

## **1 Installation**

- 2** There are two layers of insulation on top of the oven. Use a sharp knife to cut through the top layer—the soft one—using the hole in the metal as a template. Remove the soft insulation to expose the hard insulation.
- 3** The hard insulation has a precut hole that is filled with a plug. Push the plug out into the oven and discard it

### **If the location has been used before**

- 1** Remove any insulation left over from a previous detector. Check that the hole in the soft insulation conforms to the hole in the metal top plate.
- 2** If the precut hole in the hard insulation is still plugged, push the plug out and discard it.

## Install the Flow Module

**CAUTION**

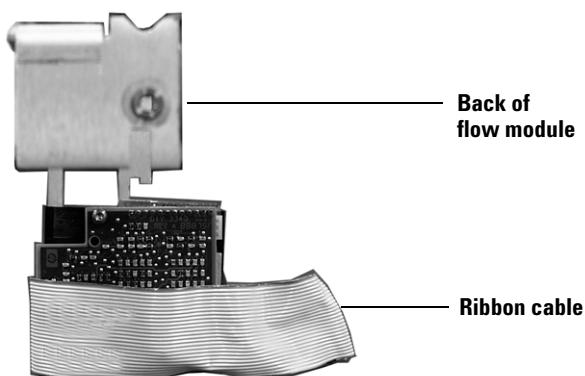
It is neither necessary nor advisable to separate detectors from their flow modules. Doing so can create leaks. Although handling the detector and flow module as a unit is awkward, it can be managed.

- 1 The tubing between the detector and flow module has been folded for shipping. Unfold the tubing, being very careful to avoid kinks or tight bends.
- 2 Locate the correct slot for the flow module (Figure 6).
  - If the detector is to be mounted in the back location, use the second slot from the left (as viewed from the back of the GC). Note that a dual-wavelength detector must be mounted in the back location.
  - If the detector is to be mounted in the front location, use the third slot from the left (as viewed from the back of the GC).

**CAUTION**

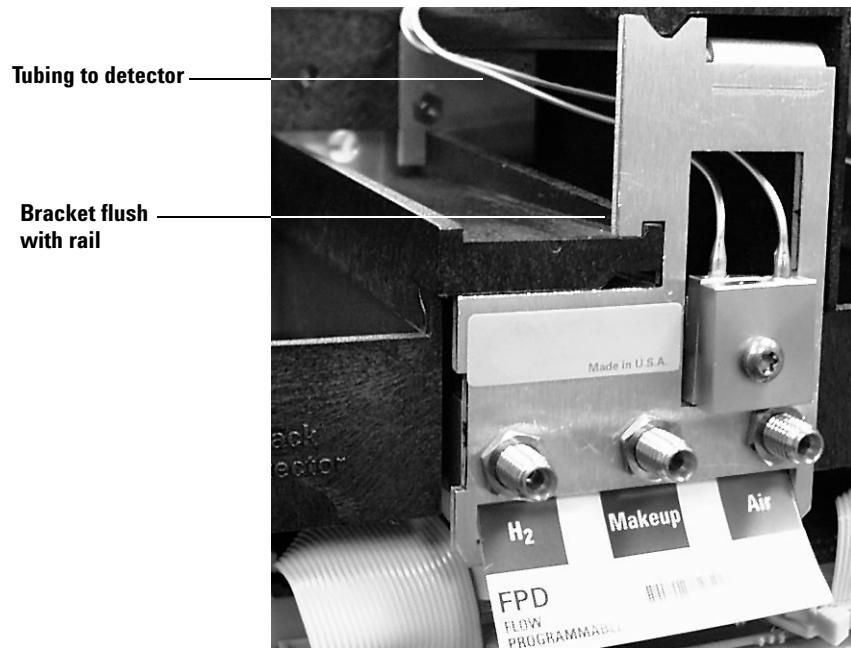
Hold the flow module by its support bracket to avoid damaging the components.

- 3 Route the ribbon cable behind the flow module assembly as shown in Figure 4. Then slide the flow module and bracket assembly into the slot until the bracket seats flush against the end of the rails (see Figure 5).



**Figure 4** Ribbon cable goes behind the flow module

## 1 Installation



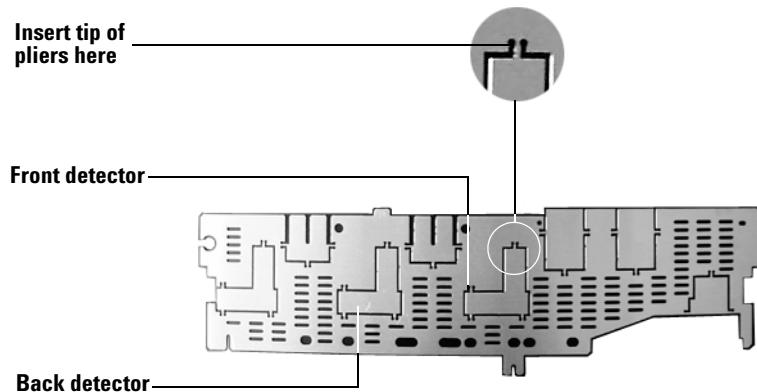
**Figure 5** Flow module installed

- 4 Connect the ribbon cable to the mating connector on the pneumatics board. The mating connector for the flow module is *not* the one directly under it. The connectors are identified by raised lettering on the plastic immediately above them.

Arrange the cable to keep it away from the valves on the right side and keep it from being pinched against the flow module.

For the back detector, you may want to loosen the flow module and slide it out of the carrier a few centimeters to connect the cable to the pneumatics board. Then, reinstall the flow module.

- a Secure the flow module in place using the T-20 Torx mounting screw on the front of the pneumatics chassis.
- b Use a pair of diagonal cutting pliers to remove the appropriate back-top panel cutout. Also remove any cutouts needed to access other flow modules or accessories installed in the GC. See Figure 6.



**Figure 6** Back-top panel cutouts

### Install the Detector

#### CAUTION

It is neither necessary nor advisable to separate detectors from their flow modules. Doing so can create leaks. Although handling the detector and flow module as a unit is awkward, it can be managed.

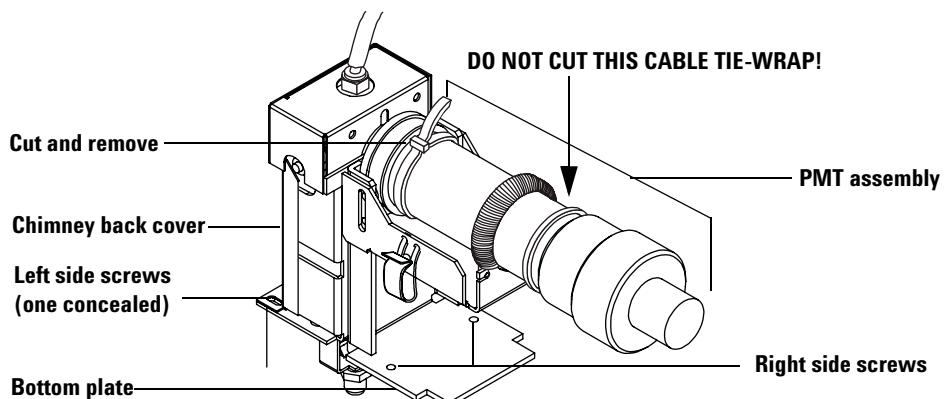
#### Detector body

- 1 Cut the shipping tie-wrap holding the PMT assembly to the bracket. Be careful not to cut any wires. See Figure 7.

#### CAUTION

There are two tie-wraps. One is the cable tie-wrap that you do **not** want to cut.

- 2 The detector body has an extended bottom plate on one side. Place the detector in the selected location with the extended plate to the right and with the column fitting passing through the insulation into the oven.
- 3 The detector is held to the oven top by four screws, one at each corner. Insert the two screws on the right but do not tighten them (see Figure 7).



**Figure 7** Installing the detector (cables removed for clarity)

- 4 Install the two screws on the left side through the chimney cover and the detector base. Press the cover against the detector and tighten the screws.
- 5 Tighten the screws on the right side.
- 6 Bend the gas lines from the flow module downward to one of the T-slots in the bracket on top of the oven. Pass the lines through the slot. Form excess length into an S-curve on top of the oven.
- 7 The FPD produces considerable water vapor when the flame is on. Connect the silicone tubing from the kit to the curved vent tube on top of the detector. Run the other end to a suitable container or drain.

## Heater/Sensor cables

### CAUTION

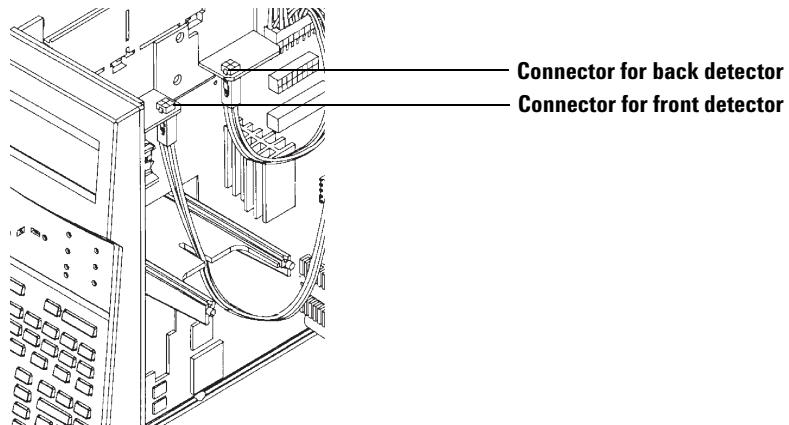
The next steps require precautions against electrostatic discharge. Use the grounded wrist strap (part number 9300-1408) and connect it to a bare metal surface of the GC. Failure to heed this caution may result in damage to the instrument or to the printed circuit board.

There are two heater/sensor cables for each detector; one for the emissions block assembly and one for the transfer line. Both cables have woven glass sleeves but one is much longer than the other.

### Short heater/sensor cable

The short heater/sensor cable plugs into a connector on the right side of the GC, as shown in Figure 8. There is only one way that the connectors will go together.

## 1 Installation

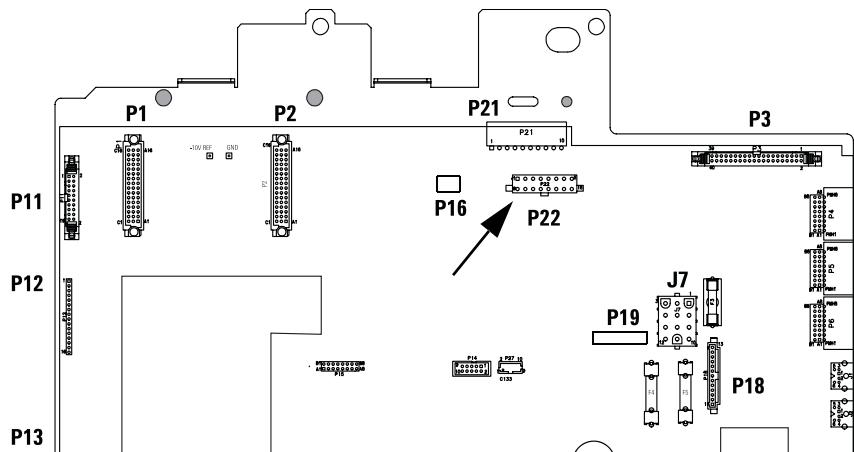


**Figure 8** Short heater/sensor cable connectors

### Long heater/sensor cable

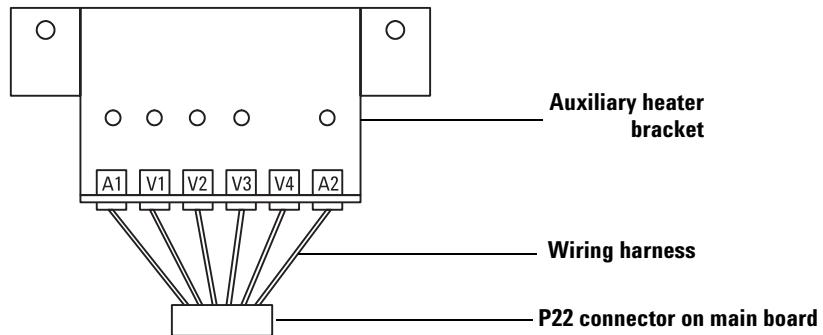
To connect the long heater/sensor cable:

- 1 Locate connector P22 on the GC main board (see the arrow in Figure 9).



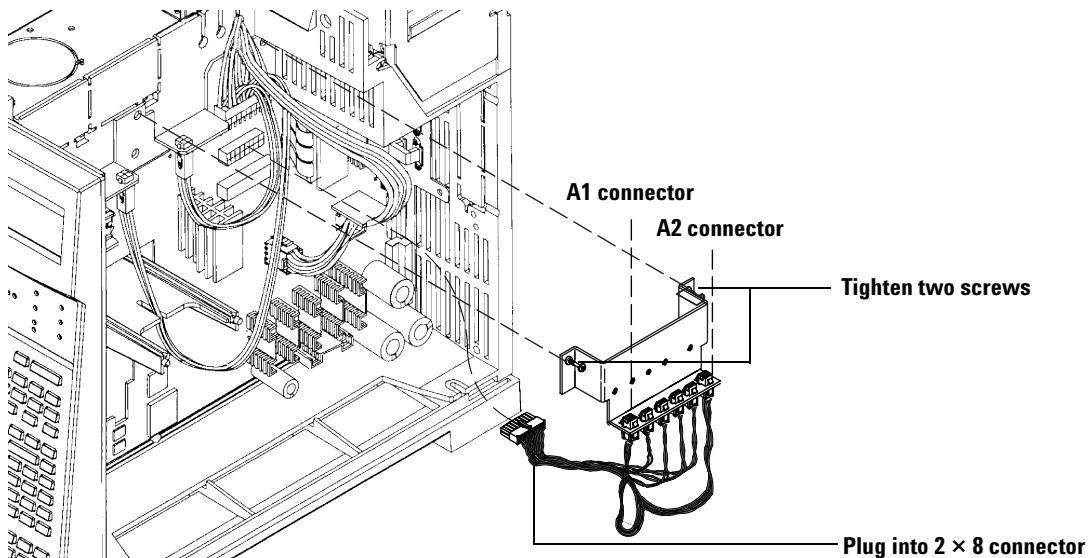
**Figure 9** Long heater/sensor cable connector

- 2** If this connector has a wiring harness and bracket attached to it (see Figure 10), skip to step 7. If nothing is attached to the connector, continue with step 3.



**Figure 10** Wiring harness and bracket

- 3** Push the wiring harness ends into the auxiliary heater bracket from the bottom as shown in Figure 11. The connectors are labeled.



**Figure 11** Attaching the assembly to the GC

## 1 Installation

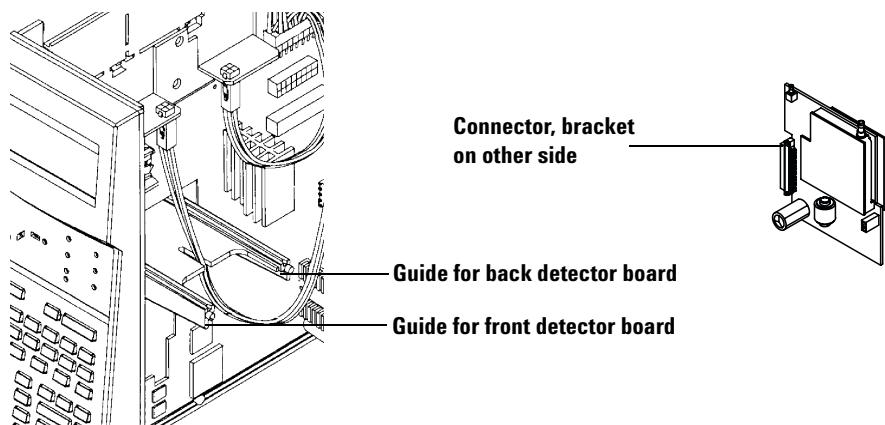
- 4 Plug the large connector on the wiring harness into the P22 connector on the main board (see Figure 9 and Figure 11).
- 5 Install the screws with the unthreaded shank into the bracket. The screws will become captured by the bracket.
- 6 Attach the bracket to the GC frame.
- 7 Pass the long heater/sensor cable under the board and plug it into a connector (see Figure 10) on the bracket as listed in Table 3.

**Table 3** Long heater/sensor cable connectors

Detector type	Location	Connector
Single-wavelength	Front	A1
Single-wavelength	Back	A2
Dual-wavelength	Back	A2

## Signal boards

- 1 Install the detector signal board(s) on the right side of the instrument behind the keyboard (see Figure 12).



**Figure 12** Installing the detector board(s)

- 2** Insert the board in the correct guide for the location used.
  - For a single-wavelength detector, the board goes in the guide for the detector position used.
  - For a dual-wavelength detector, boards go in both guides. The board in the back slot becomes the master board, providing high voltage and signal processing for one PMT assembly and ignitor power for the detector emissions block assembly. It controls the other board.
  - The board in the front guide provides high voltage and signal processing for the second PMT assembly.
- 3** Press the board firmly into place so that the connector on the board engages the matching connector on the GC main circuit board.
- 4** Secure the board with the captive screw in the bracket.

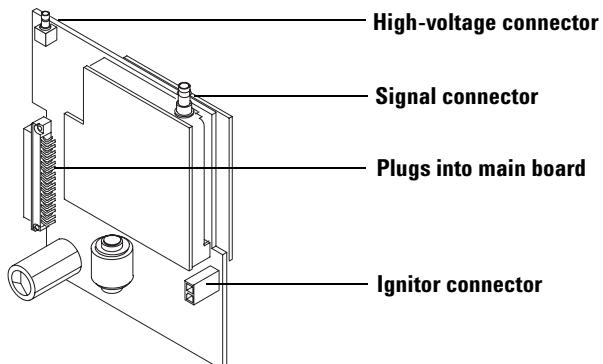
## Signal board cable connections

### CAUTION

With a dual-wavelength FPD, it does not matter which of the two circuit boards is used for which PMT (see Figure 12). However, the high voltage and signal cables from each PMT must go to the same board. Do not “cross-wire.”

- 1** Connect the cables to the signal boards. Collect the cables and route them through the metal clip on the side of the PMT assembly support.

Figure 13 locates the detector board connectors.



**Figure 13** Detector board connector locations

### High voltage cable

The high voltage cable is black with a chrome connector on the end (Figure 14). Press it down over the high voltage connector on the board until it clicks into place.



**Figure 14** High voltage cable

### Signal cable

The signal cable is black with a BNC connector on the end (Figure 15). Press it onto the signal connector on the board and turn the knurled ring to lock it in place.

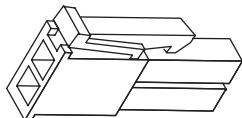


**Figure 15** Signal cable

### Ignitor cable

The ignitor cable is gray and ends in a 2-pin flat connector (Figure 16). Press it into the ignitor connector on the board until it locks into place. There is only one way that it can be connected.

If you are installing a dual-wavelength detector, there will be only one ignitor cable. Connect it to the board in the back detector slot.



**Figure 16** Ignitor connector

## Update the Firmware

### 6890A and 6890 Plus

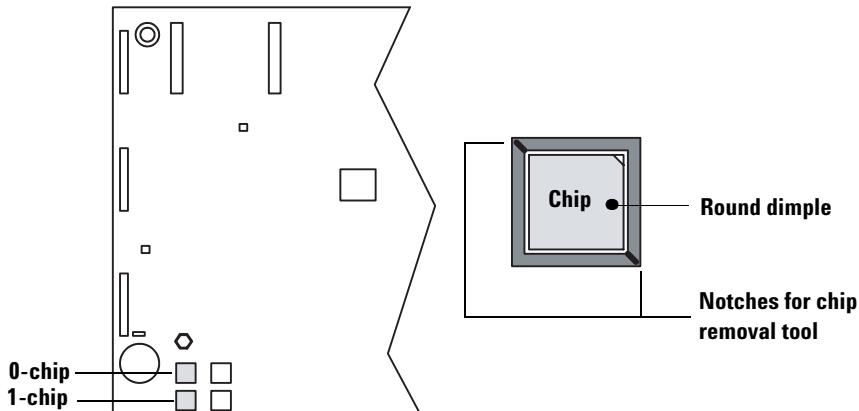
If the present firmware is earlier than A.03.08, you must replace the chips with those included in the kit. These chips contain firmware version A.03.08.

#### NOTE

The GC displays the current firmware version when you first turn it on or press [STATUS] followed by [Clear].

To replace the chips:

- 1 Examine the right side of the GC. On the main board, just below the large cutout on the left, are four chip sockets (see Figure 17). The two on the left contain the operating programs (firmware) that run the GC.



**Figure 17** Chip sockets

The chips that came in the kit are identified by a part number, a version number, and a suffix of either ".0" or ".1". They are referred to as the 0-chip and the 1-chip, respectively.

## 1 Installation

### CAUTION

This procedure requires precautions against electrostatic discharge. Use a grounded wrist strap (part number 9300-1408) and connect it to a bare metal surface of the GC. Failure to heed this caution may result in damage to the instrument.

---

- 2** Use a chip removal tool with a gentle rocking/pulling motion to remove a chip from the socket.

### CAUTION

Be sure to note which chip you are removing. You want to be sure to place the correct replacement chip in the correct socket.

---

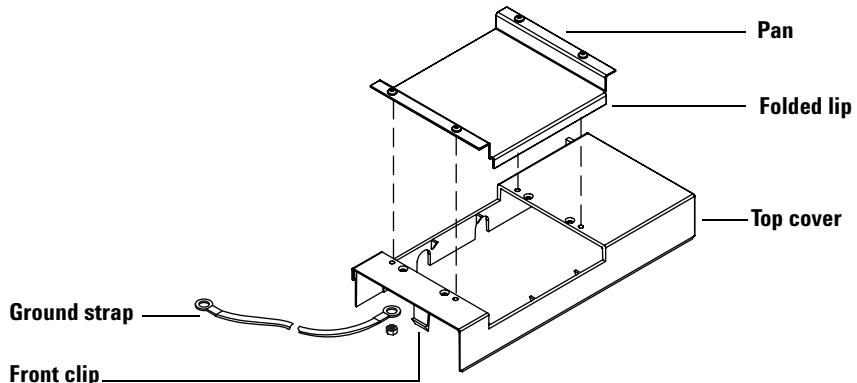
- 3** Orient the chip so the round dimple is at the right. When the chip is properly aligned, snap it into place in the socket indicated in Figure 17 using firm pressure.
- 4** Repeat step 2 and step 3 for the other chip.

## 6890N

To download the latest 6890N firmware, visit the Agilent Web Site at [www.agilent.com/chem](http://www.agilent.com/chem), login or register, and navigate to firmware.

## Replace the Covers and Panels

- 1 Place the back-top panel on its left-most mounting screw. Use the screw as a hinge and angle the panel while sliding each ID tag through its cutout in the panel, working from left to right. When all the tags are through the panel, finish installing the panel on the GC.
- 2 Re-install the RFI cover, the pneumatics chassis cover, and the top cover.
- 3 Re-install the right-side panel. Be sure that the hook at the back-top corner is engaged.
- 4 The electronics cover that came in the kit is in two parts (see Figure 18).



**Figure 18** The electronics cover

- a Unscrew the four captive screws and remove the pan.
- b Align the base and lower it into place. Two clips secure it.
- c Slide the pan—folded lip to the right—under the PMT assemblies).
- d Attach the groundstrap under the top cover as shown in Figure 18. Connect it between one of the pan mounting screws and one of the screws that secure the detector to the top of the oven.
- e Align the other screws with the holes and tighten all four.

## **Return the GC to Operating Condition**

- 1** Restore power.
- 2** Turn off the flame to prevent condensation in the detector.
- 3** Restore gas flows and pressures and check for leaks. See the GC user information for leak testing information.
- 4** Reboot the GC. As it is turning back on, check to see if it detects that it has an FPD. If not, check these connections:
  - Detector card to main board
  - Flow module to pneumatics board
  - Heater/Sensor connections
  - Ignition cable to detector card
  - High-voltage cable to detector cards
  - Signal cable to detector card
  - Correct ROM installed on main board

If you have temperature control for the detector but not flow control, check the connection between the flow module and the pneumatics board.

## **2**

# **Checkout Procedure**

Overview	36
Creating Methods	38
Phosphorus method	38
Sulfur method	40
Using ChemStation macros	41
Running the Tests	42
Interpreting Results	46

When you have installed the FPD, you should test it to verify that it works correctly. This chapter describes a test protocol for running sulfur and phosphorus samples through the GC and examining the results.

The protocol will differ depending on the equipment you have. This chapter describes the protocol in terms of a ChemStation, but contains sufficient information so that the tests can be run with just a strip-chart recorder.



### Overview

**NOTE**

This procedure assumes that the tester is familiar with the safe operation and maintenance of the gas chromatography system, including the automatic liquid sampler, GC, and data handling device.

---

This is the recommended sequence of steps for running the checkout. Install the column.

- 1 Create and save the phosphorus and sulfur methods.
- 2 Load the phosphorus method, turn off the flame and electrometer.
- 3 Install the phosphorus filter.
- 4 Set inlet, oven, and detector to 250 °C to bake out for 15 minutes.
- 5 Reload the phosphorus method. Monitor signal output. This output typically runs between 40 and 55 but can be as high as 70.
- 6 When it levels off, run one sample. Interpret the results (page 46).
- 7 Turn off the electrometer. Install the sulfur filter and spacer.
- 8 Load the sulfur method. Monitor signal output. The output typically runs between 50 and 60 but can be as high as 70.
- 9 When it levels off, run one sample. Interpret the results (page 46).

### Materials needed

- Column: 5% Phenyl Methyl Siloxane Capillary 30.0 m × 0.32 mm × 0.25 µm (part number 19091J-413).  
For Japan only: HP-5MS 30 m × 0.32 mm × 0.50 µm (part number 19091S-113).
- Checkout sample, part number 5188-5953.  
For Japan only, part number 5188-5245.
- For cool on-column inlet use: 5-mm septa (part number 5183-4758, 50/pk), 5-µL on-column syringe for 320-µm column (part number 5182-0836), and needle for 320-µm column (5182-0831).

- For split/splitless inlet use: 11-mm septa (part number 5183-4757 50/pk), 10- $\mu$ L syringe (part number 5181-1267), or equivalent, and a liner. The liner can be general purpose (part number 5183-4711) or single-taper deactivated with (part number 5062-3587) or without (part number 5181-3316) glass wool.

For Japan only: 11-mm septa (part number 5183-4757 50/pk), 10- $\mu$ L syringe (part number 5181-1267), or equivalent, and a single-taper liner deactivated without (part number 5181-3316) glass wool.

- Chromatographic-grade iso-octane for syringe wash solvent.
- Chromatographic-grade 99.9995% purity gas: helium as carrier, nitrogen as makeup, hydrogen and air.
- 4-mL solvent and waste bottles or equivalent for autoinjector.
- 2-mL sample bottles or equivalent for sample.

## 2 Checkout Procedure

### Creating Methods

Create the test methods using these steps:

- 1 Create a phosphorus method (Table 4).
- 2 Save it using a name that identifies it as the phosphorus method (such as **P\_Ckout.m**).
- 3 Make a copy and modify the detector flow parameters in the copy to change it to a sulfur method (page 40).
- 4 Save the copy using a distinctive name that identifies it as a sulfur method (such as **S\_Ckout.m**).

#### Phosphorus method

Table 4 contains the parameters for the phosphorus method. Give the method a meaningful name that allows you to distinguish it from the sulfur method.

**Table 4** Phosphorus checkout method default parameters value

Parameter/Setting	Sample 5188-5953	Sample 5188-5245
<b>Oven</b>		
Initial temp, °C	70	70
Initial time, min	0.00 min	0.00 min
Rate 1, °C/min	25.00	10.00
Final temp 1, °C	150	105
Final time 1, min	0	0
Rate 2, °C/min	5	20
Final temp 2, °C	190	190
Final time 2, min	4	1
<b>Column</b>		
	Agilent 19091J-413, HP5, 30.0 m × 320 µm × 0.25 µm	Agilent 19091S-113, HP5 MS, 30.0 m × 0.32 mm × 0.50 µm
Mode	Constant pressure	Constant pressure
Pressure	25 psi	25 psi
<b>Detector</b>		
Temperature, °C	200 (On)	200 (On)
Hydrogen flow	75.0 mL/min (On)	75.0 mL/min (On)

**Table 4** Phosphorus checkout method default parameters value (continued)

<b>Parameter/Setting</b>	<b>Sample 5188-5953</b>	<b>Sample 5188-5245</b>
Air (Oxidizer) flow	100.0 mL/min (On)	100.0 mL/min (On)
Mode	Constant makeup flow OFF	Constant makeup flow OFF
Makeup flow	60.0 mL/min (On)	60.0 mL/min (On)
Makeup gas type	Nitrogen	Nitrogen
Flame	On	On
Lit offset	2.00	2.00
Electrometer	On	On
<b>Signal</b>		
Data rate	5 Hz	5 Hz
Type	Front or back detector	Front or back detector
Save data	On	On
Zero	0.0 (Off)	0.0 (Off)
Range	0	0
Fast peaks	Off	Off
Attenuation	0	0
<b>Column Comp 1</b>	Derive from detector	Derive from detector
<b>Inlet</b>		
Temperature, °C	200 (Split/Splitless inlet, and see below) Oven track (Cool On-Column inlet)	250 (Split/Splitless inlet, and see below) Oven track (Cool On-Column inlet)
Init pressure	25.00 psi (On)	25.00 psi (On)
Gas saver	Off	Off
<b>Split/Splitless inlet</b>		
Mode	Splitless	Splitless
Total flow	69.5 mL/min	69.5 mL/min
Purge flow to split vent	50 mL/min @ 0.75 min	50 mL/min @ 0.75 min
<b>Injector (if installed)</b>		
Sample washes	2	2
Sample pumps	8	8
Injection volume	1.0 µL	1.0 µL
Syringe size	5.0 or 10 µL	5.0 or 10 µL

## 2 Checkout Procedure

**Table 4** Phosphorus checkout method default parameters value (continued)

Parameter/Setting	Sample 5188-5953	Sample 5188-5245
PreInj Solvent A Washes	0	0
PreInj Solvent B Washes	0	0
PostInj Solvent A Washes	4	4
PostInj Solvent B Washes	4	4
Viscosity delay	OFF	OFF
Plunger speed	Fast	Fast
PreInjection dwell	0.00	0.00
PostInjection dwell	0.00	0.00
<b>Sample</b>	part number 5188-5953	part number 5188-5245
Injection volume	1.0 µL	1.0 µL

Additional settings applicable to Agilent data systems are listed in Table 5. The settings available are for the GC ChemStation. Other data systems vary.

**Table 5** Additional settings for Agilent Data Systems

Parameter/Setting	Value
<b>Report settings</b>	
Report style	Short or None
Destination	None

## Sulfur method

Make a copy (Save As in ChemStation) of the phosphorus method. Rename the copy to indicate that it is a sulfur method. Edit it as indicated in Table 6.

**Table 6** Sulfur method parameters

Parameter	Value
H <sub>2</sub> flow	50 mL/min
Air flow	60

## Using ChemStation macros

There are two macros designed for ChemStation that you use to interpret and analyze the data collected from the sulfur and phosphorus tests. Table 7 lists the macros. They can be downloaded from [www.chem.agilent.com/cag/servsup/usersoft/files/ChemStation\\_Macros\\_Collection.htm](http://www.chem.agilent.com/cag/servsup/usersoft/files/ChemStation_Macros_Collection.htm). Look for Data Systems, then Downloads and Utilities.

**Table 7** ChemStation macros

Macro name	Description
MEPCK_S.MAC Japan only: fpds.xls.mac	Processes data collected from sulfur tests.
MEPCK_P.MAC Japan only: fpdp.xls.mac	Processes data collected from phosphorus tests.

These macros calculate the area counts, MDL, and noise, and print a report to the printer setup in the ChemStation. The report includes the chromatogram, the noise plot, and several calculations.

Place the macros in the appropriate subdirectory (Table 8) where ChemStation can find them.

**Table 8** Macro subdirectories

Software rev.	Macro location	Summary result log location
B.01.xx or later	CHEM32\CORE	CHEM32\n*
A.10.xx or earlier	HPCHEM\CORE	HPCHEM\n

\* n is the instrument number.

The macro can be set to run automatically as part of the method as a post-run program, or can be run on a loaded data file from the ChemStation Data Analysis view.

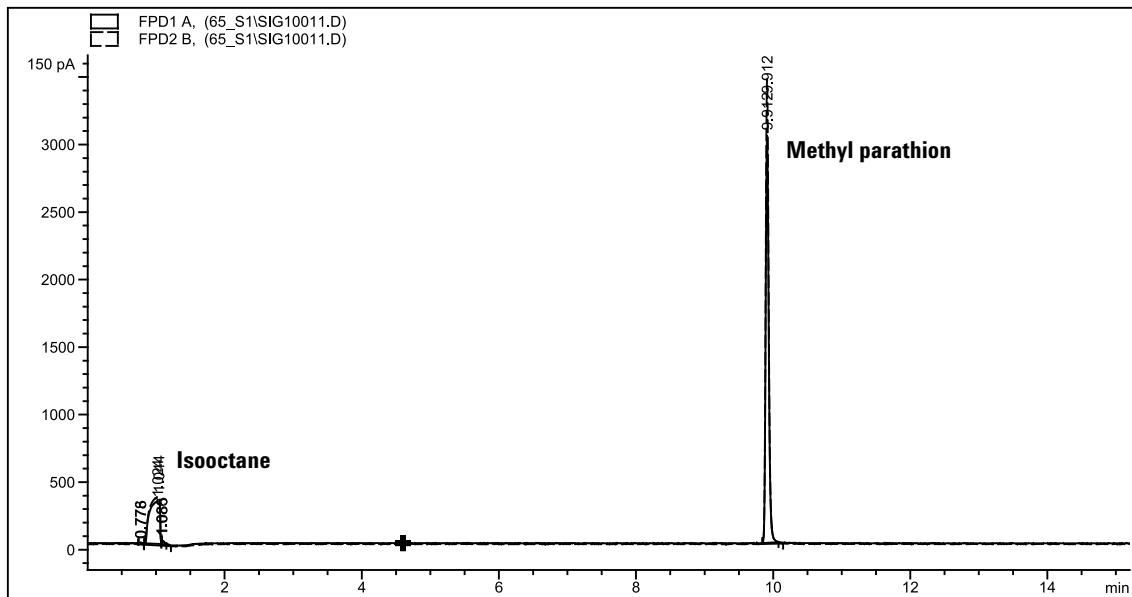
## 2 Checkout Procedure

### Running the Tests

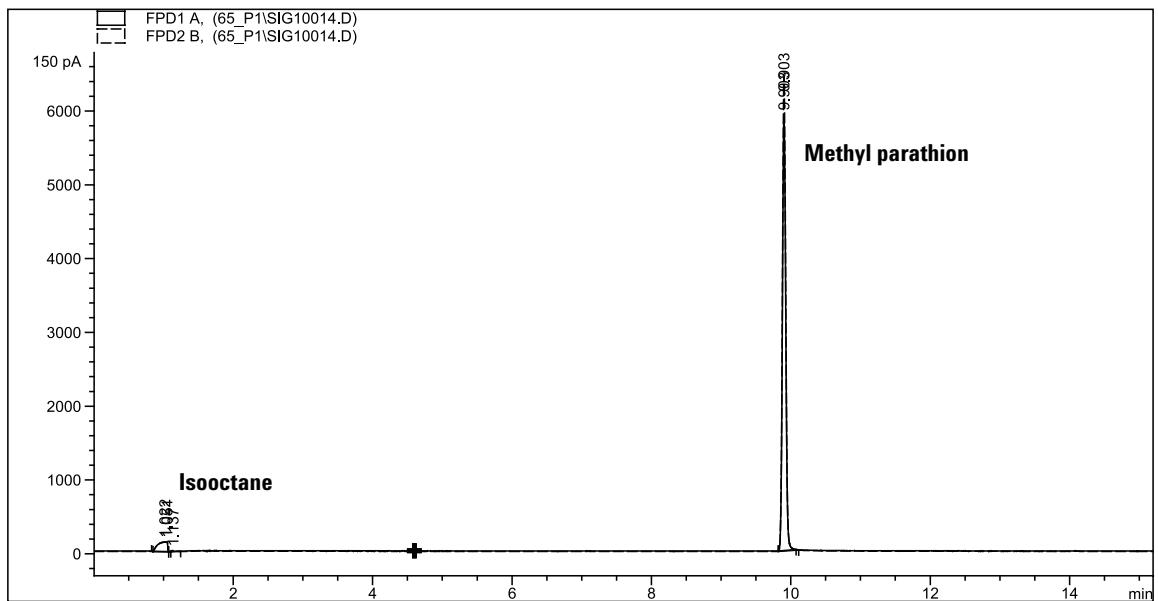
After setting the conditions, wait for the baseline to stabilize (about 2 hours) before running samples.

Figure 19 and Figure 20 show typical online plots for sulfur and phosphorus tests using the checkout methods described in “Creating Methods” on page 38.

Figure 21 and Figure 22 show the same plots for the Japan-only sample.

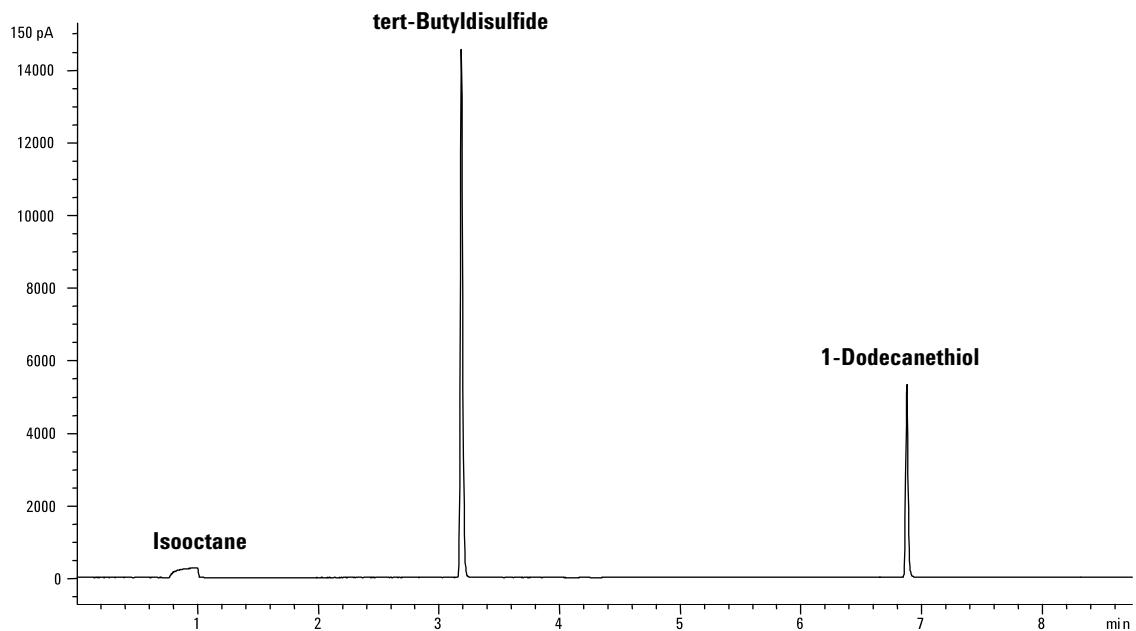


**Figure 19** Typical chromatogram for sulfur tests, sample 5188-5953

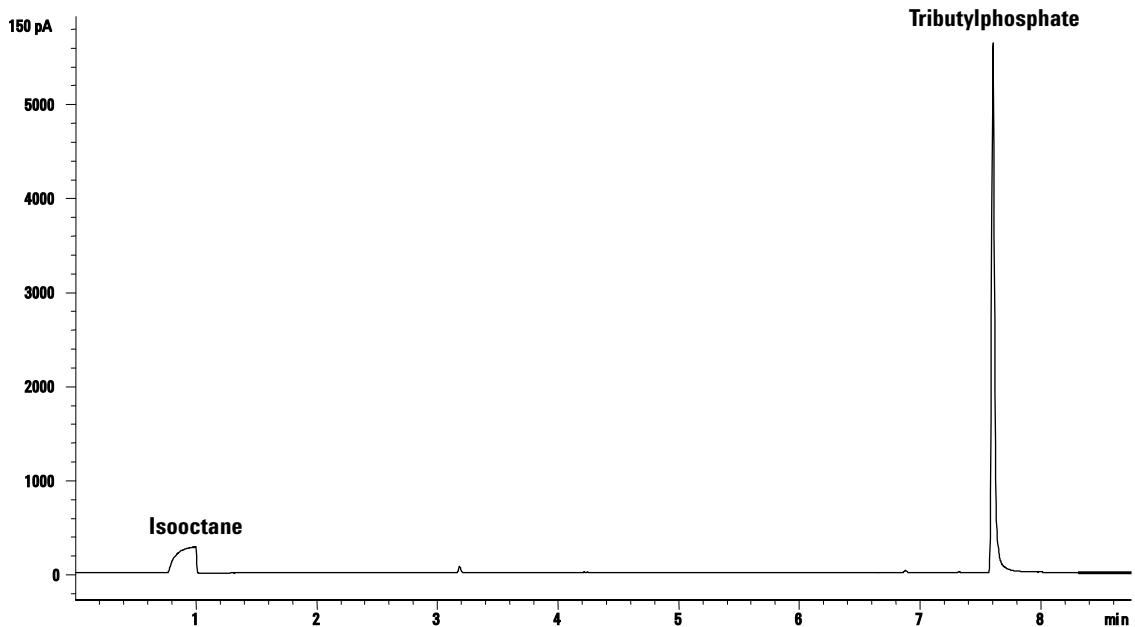


**Figure 20** Typical chromatogram for phosphorus tests, sample 5188-5953

## 2 Checkout Procedure



**Figure 21** Typical chromatogram for sulfur tests, sample 5188-5245 (Japan only)



**Figure 22** Typical chromatogram for phosphorus tests, sample 5188-5245 (Japan only)

### CAUTION

If you receive a message like the following, check your printer setup and make any corrections.

... has problems. , with page file:  
C:\WINNT\TEMP\~P3D042A.TMP  
**Initial printing problem, 202.**  
**System resources are low and/or device has problems.**

### NOTE

Depending on the column length, the sulfur chromatograph may contain a contaminant peak during the noise measurement. Reprocess the run by loading the signal and typing **macro mepck\_s.mac,go,3**

in the command line. This starts the noise measurement at 3 minutes instead of the default 3.8 minutes.

## **2 Checkout Procedure**

# **Interpreting Results**

Compare the reported output against the values listed in Table 9 and Table 10. Your value should be within the limits listed.

## **For phosphorus**

**Table 9** Evaluating checkout runs

<b>FPD P filter</b>	<b>Typical range after 24 hours</b>	<b>Limits at installation</b>
MDL (pg/sec)	0.06 to 0.08	≤0.10
Peak area	19000 to 32000	≥19000
Signal height	5000 to 11000	—
Noise	1.6 to 3.0	≤4
Half-width (min)	0.05 to 0.07	—
Signal offset	34 to 80	≤80

## **For sulfur**

**Table 10** Evaluating checkout runs

<b>FPD P filter</b>	<b>Typical range after 24 hours</b>	<b>Limits at installation</b>
MDL (pg/s)	3.8 to 5	≤6
Peak area	8000 to 19000	≥8000
Signal height	2500 to 6000	—
Noise	2 to 4	≤5
Half-width (min)	0.06 to 0.08	—
Signal offset	34 to 65	≤70

## 3 Operation

General Rules 48  
Installing Columns 50

This chapter describes **new or changed** operating information (at the time of this printing) that updates the information found in the 6890 user documentation. **Refer to the GC user information for any topics not covered here.**



## General Rules

### Starting up

The FPD creates a great deal of water vapor when the flame is on. This could condense in the vent tube on top of the detector and drop onto the flame, possibly extinguishing it. To avoid this, turn the heaters on, wait 20 minutes for the vent to heat up, and then ignite the flame. Water vapor will now make it over the top of the vent tube before condensing.

### Shutting down

For similar reasons, extinguish the flame before turning the heaters off.

### Photomultiplier (PMT) protection

The PMT is extremely sensitive to light. Always turn the electrometer off (which turns off the high voltage to the PMT) before removing the PMT assembly or opening the emissions block assembly. Failing to do this can destroy the PMT.

Even with the electrometer off, protect the PMT from room light. Cap the housing when removed, place it end down to exclude light, reduce room light level before exposing the PMT, and so on. A brief exposure (always with the electrometer turned off) will not damage it but prolonged exposure will cause a gradual loss of sensitivity.

### Lit Offset

The default Lit Offset is 2.0 pA.

### High-purity gases

High-purity gases have a lower sulfur content. Standard purity gases have a higher sulfur content which impairs sulfur detection in the compound being studied. Instrument or Chromatographic grades work well.

Agilent recommends using helium carrier, nitrogen makeup gas, and air with 99.9995% purity or better. Use hydrocarbon, oxygen, and moisture traps. Select traps to remove sulfur compounds from detector air and nitrogen gases. A helium getter is also recommended.

**Inlet liners**

Compounds containing sulfur may adsorb to an inlet liner and degrade the GC's performance. Use deactivated, clean liners or a cool on-column inlet, which injects directly onto the column.

For best results with splitless injection, use liner 5181-3316.

## Installing Columns

### Capillary columns

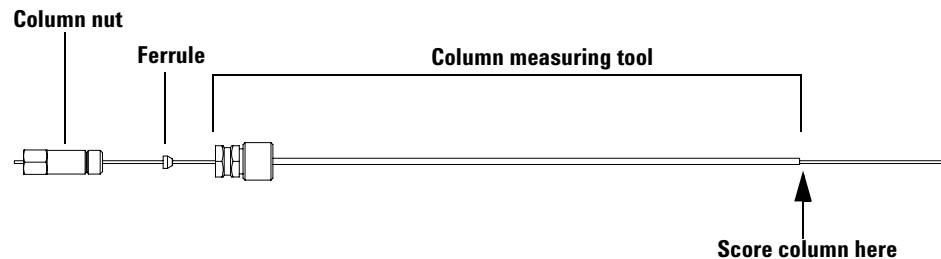
#### CAUTION

Wear safety glasses while handling, cutting, or installing fused silica capillary columns. To prevent puncture wounds, use care when handling these columns.

The following procedure ensures that the FPD works efficiently. A special tool is used to cut the column to the correct length.

#### Swage the column ferrule

- 1 Install a column nut and ferrule on the end of the column.
- 2 Insert the end of the column through the column measuring tool (19256-80640) so that the end protrudes beyond the tool (Figure 23).

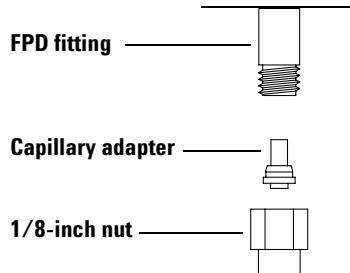


**Figure 23** Cutting the column to length

- 3 Tighten the column nut until it grips the column. Use a pair of wrenches to tighten it an additional 1/8 to 1/4 turn.
- 4 Use a wafer cutter at 45° to score the column.
- 5 Snap off the column end.
- 6 The column may protrude 1 to 2 mm beyond the end of the tool.
- 7 Remove the column, nut, and swaged ferrule from the tool.

### Install the column

- 1 Install the capillary adapter in the detector fitting (Figure 24).



**Figure 24** Capillary adapter

- 2 Carefully thread the swaged column up into the adapter. Finger-tighten the column nut, then tighten an additional 1/8-turn with a wrench.

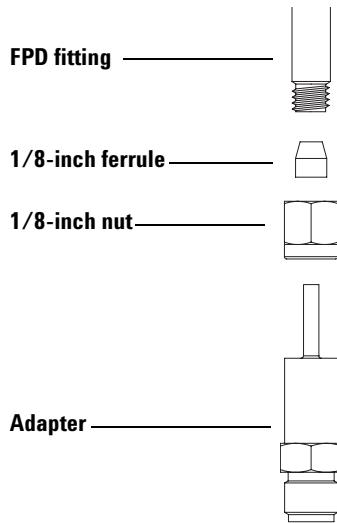
### Packed columns, 1/8-inch

A Type B 1/8-inch column connects directly to the fitting on the bottom of the FPD. No adapter is required.

### 3 Operation

#### Packed columns, 1/4-inch

- 1 Install the adapter in the detector fitting (Figure 25). Place the nut and ferrule on the adapter, insert the adapter into the FPD fitting, and tighten the nut.



**Figure 25** 1/4-inch column adapter

- 2 Connect the 1/4-inch column to the adapter in the usual way. Use Type B glass columns.

## **4**

# **Maintenance and Troubleshooting**

- Changing the Wavelength Filter 54
- Accessing Heaters and Ignitor 58
- Replacing the Heater/Sensor Assemblies 62
- Replacing the Ignitor 69
- Replacing the Inert Transfer Line 73
- Troubleshooting 76

This chapter describes maintenance procedures that can be performed by an FPD user. The troubleshooting section is a guide to frequently found things, including hardware, software, and operational, that can go wrong with suggestions on how to correct them.



### Changing the Wavelength Filter

Changing the wavelength filter takes about 5 minutes. It requires you to turn off the electrometer, remove the photomultiplier tube assembly, and change the filter.

#### CAUTION

Do not touch the filters with your bare hands. For optimum performance and to avoid scratches, use lint free gloves for assembling and inserting the filters into the assembly.

---

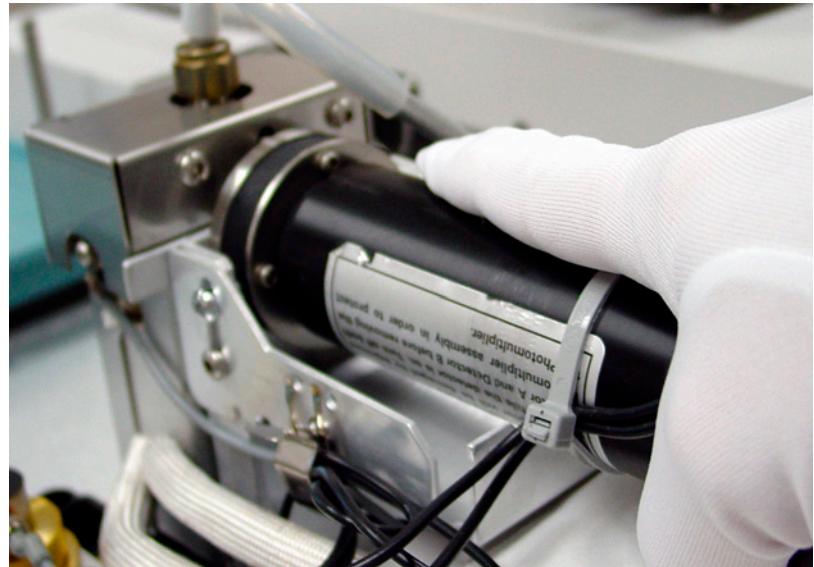
#### Materials needed

- 1000-1437 Sulfur filter and 19256-20910 filter spacer
- 19256-80010 Phosphorus filter
- 5080-5400 Cotton swabs
- Toothpick or cotton swab
- Lens tissue
- 8650-0030 (large) or 8650-0029 (small) Nylon lint-free gloves

#### Procedure

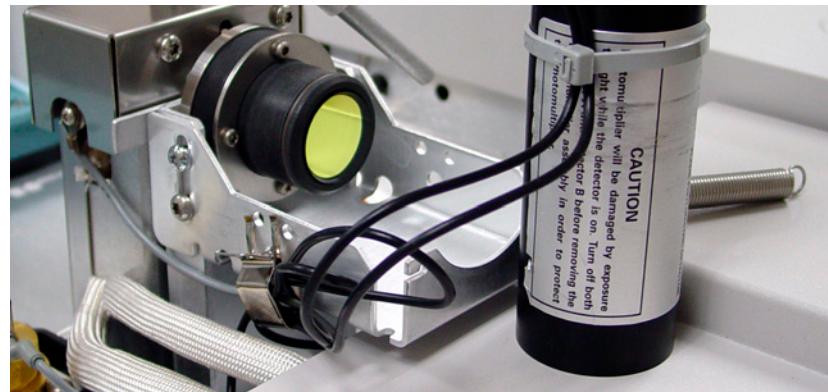
- 1 Turn off the detector electrometer.

- 2 Disconnect the retaining spring that holds the PMT assembly to the bracket. With a rotating motion, pull the assembly away from the filter housing (Figure 26).



**Figure 26** Removing the PMT assembly

- 3 To prevent light from damaging the PMT, immediately cap the end or place it face down (Figure 27).



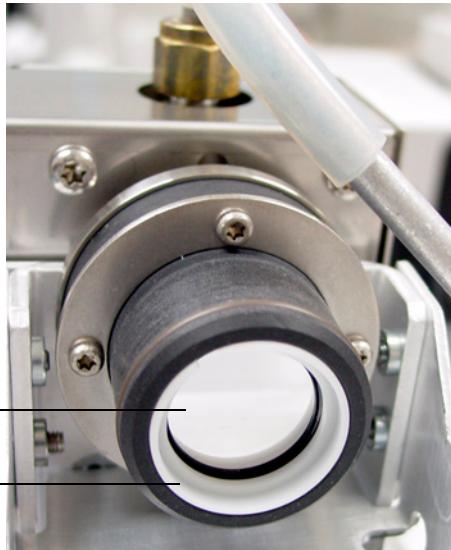
**Figure 27** Protecting the PMT

## 4 Maintenance and Troubleshooting

- 4 Place a clean cloth under the filter housing to catch the filter.

**Phosphorus filter:** use the sharpened wooden tip of a toothpick or cotton swab to dislodge the filter from the housing.

**Sulfur filter:** (Figure 28) use the wooden tip of the cotton swab to remove the filter spacer. Then remove the filter as above.



**Figure 28** Sulfur filter and spacer

- 5 Use lens tissue to clean the new filter. Install it in the filter housing. If you are installing a sulfur filter, add the filter spacer.

### CAUTION

Do not use cleaning fluids. Cleaning fluids will damage lens coatings.

### CAUTION

Filters are designed for the light from the flame to pass through in a specific direction. On the edge of the phosphorus filter, there is a triangle. On the edge of the sulfur filter, there is an arrow. These should face away from the flame and toward the PMT.

- 6** Replace the PMT assemblies and secure them with the springs.
- 7** Restore the operating conditions.

### Accessing Heaters and Ignitor

Removing the vent and cover and, on the dual-wavelength detector, the left PMT assembly to access the heaters or ignitor takes about 5 minutes. It requires you to turn off the GC.

#### CAUTION

When turning the GC off, turn off the flame first to prevent condensation from dripping into the jet and column.

---

You may wish to access this area for the following reasons:

- Replace the ignitor.
- Replace seals and O-rings.
- Replace the transfer line assembly.
- Replace or check the heater and PRT sensors.

### Materials needed

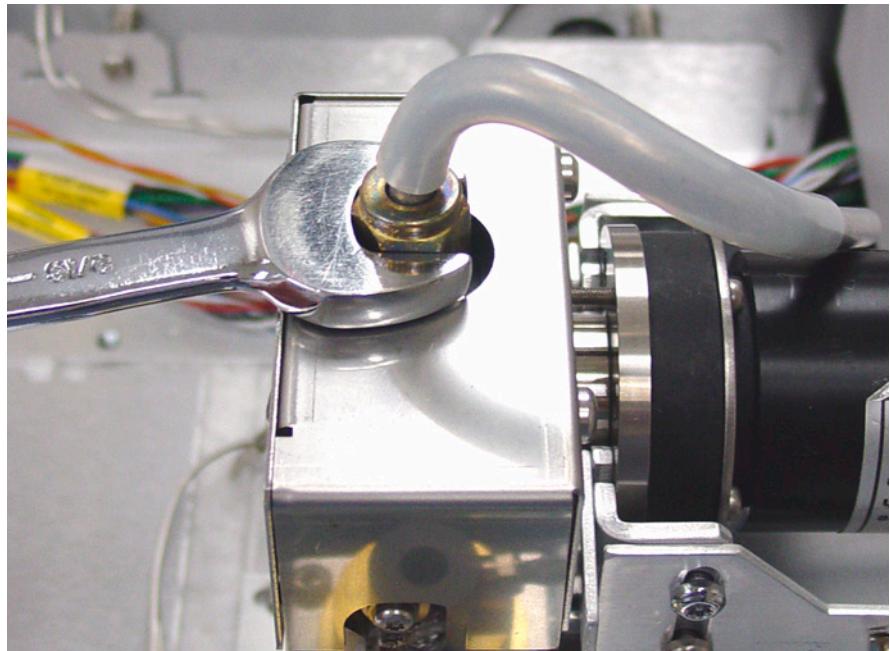
- 8710-0803 9/16-inch wrench
- 8710-1807 T-20 Torx driver

### Procedure

#### To remove the vent tube and cover

- 1 Turn off the flame.
- 2 Turn off the GC.
- 3 Wait until the detector cools.

- 4 Use a wrench to loosen and remove the vent tube assembly (Figure 29).



**Figure 29** Removing the vent tube

- 5 **Single-wavelength detector.** Use a Torx T-20 driver to remove the screws securing the FPD cover. There are two screws at the bottom of the left side (Figure 30) and two screws at the top of the right side (Figure 31).

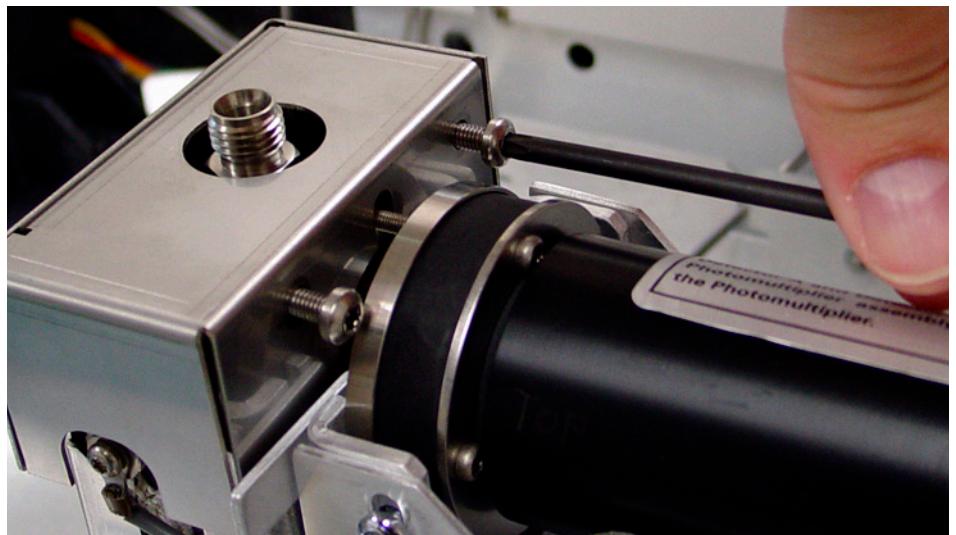
**Dual-wavelength detector.** Use a Torx T-20 driver to remove the screws securing the FPD cover. There are two screws at the top of the right side (Figure 31).

Lift the cover off the detector.

## 4 Maintenance and Troubleshooting



**Figure 30** Cover screws, left side (single-wavelength FPD)



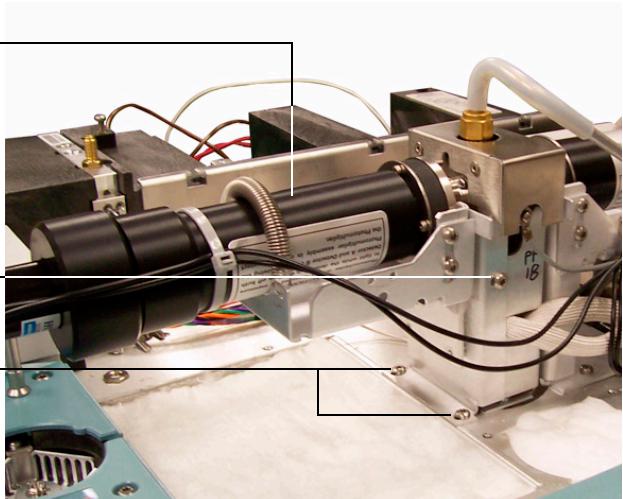
**Figure 31** Cover screws, right side (both FPDs)

**6 Dual-wavelength detector only.** Remove the four screws securing the Dual FPD chimney front (with the attached dual main bracket and PMT

assembly). Remove the entire left part of the detector. Follow the order of operations in Figure 32. Cover the open end of the PMT assembly to protect the tube from light.

**3. Remove PMT assembly and attached parts.**

- 2. Remove two screws (second screw is on rear side of chimney).**
- 1. Remove two screws.**



**Figure 32** Removing the left PMT assembly

**To reinstall the vent tube and cover**

- 1** Single-wavelength detector
  - a** Start the two screws on the right side of the cover.
  - b** Start and tighten the screws at the base on the left side.
  - c** Tighten the screws on the right side.
- 2** Dual-wavelength detector
  - a** Place and support the PMT assembly so that the screw holes line up.
  - b** Install and tighten the four screws.
  - c** Install the cover (two screws).
- 3** Replace the vent tube assembly.
- 4** Turn on the GC. Confirm that the flame is off.
- 5** Restore the operating conditions.
- 6** Wait 20 minutes for the detector to heat up, then ignite the flame.

### Replacing the Heater/Sensor Assemblies

Replacing the heater/sensor assemblies takes about 30 minutes. It requires you to turn off the GC, remove the vent, the detector cover, and, on the dual-wavelength detector, the left PMT assembly.

#### CAUTION

When turning the GC off, turn off the flame first to prevent condensation from dripping into the jet and column.

You may wish to replace or check the FPD heater/sensor assemblies for the following reasons:

- One or both of the heaters or sensors are defective.
- The actual temperature reading on the display of the heaters is cycling more than 1 °C.

#### Materials needed

- G1535-60610 Heater/Sensor assembly with short lead for the emissions block assembly.
- G1535-60620 Heater/Sensor assembly with long lead for the transfer line.
- ESD protection: For example, a wrist strap
- Wrenches: 8710-0803 (9/16-inch), 8720-0010 (5/8-inch), 1/4-inch × 3/8-inch
- Torx drivers: 8710-2140 T-10 and 8710-1807 T-20.

#### Procedure

- 1 Turn off the flame.
- 2 Turn off the GC.
- 3 Remove the vent assembly and cover. Refer to “Accessing Heaters and Ignitor” on page 58.
- 4 Put on an ESD wrist strap. Remove the right-side cover to access the GC electronics. Disconnect the heater/sensor leads from the auxiliary heater board and the connectors above the main board.
- 5 Carefully pull the cables up onto the top of the GC.

**Transfer line**

- 1 Use a Torx T-10 driver to remove the screw and retainer clip holding the lower heater/sensor assembly (Figure 33). Remove the heater and sensor from the transfer line.



**Figure 33** Transfer line heater/sensor

- 2 Remove the protective cap from the temperature sensor of the heater/sensor assembly with the short cable.

## 4 Maintenance and Troubleshooting

- 3 Insert the heater and sensor into the transfer line. Make sure the sensor is seated at the bottom of the hole. If not, the AUX temperature will wander above and below the detector setpoint.



**Figure 34** Install the transfer line heater/sensor

- 4 Position the retainer clip over the heater/sensor assembly and install the screw.



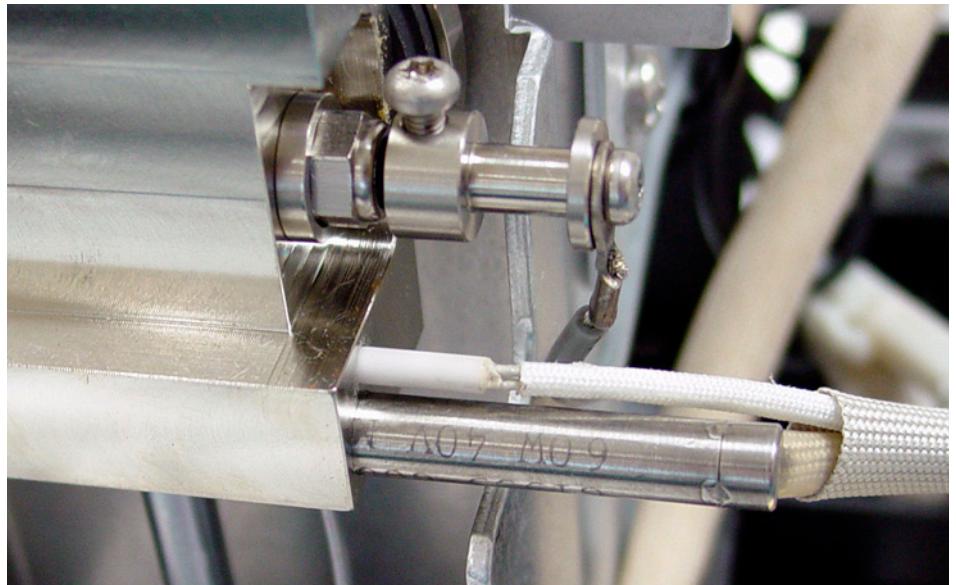
**Figure 35** Install the retainer clip

### Emissions block assembly

- 1 Remove the upper heater and temperature sensor from the emissions block assembly.
- 2 Remove the protective cap from the sensor of the heater/sensor assembly with the long cable.

## **4 Maintenance and Troubleshooting**

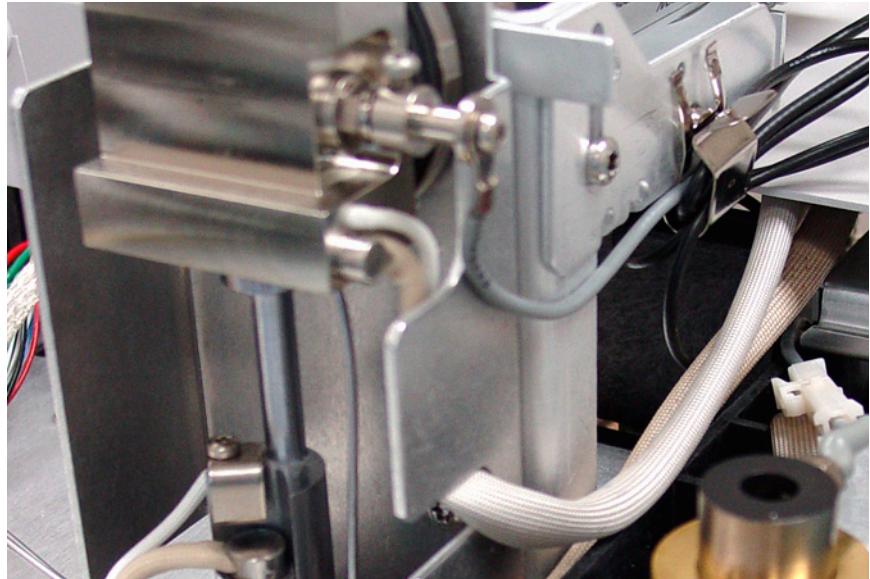
- 3** Install the upper heater and sensor in the emissions block assembly.



**Figure 36** Install the upper heater and sensor

### Closing up

- 1 Route the heater/sensor cables out of the bracket (Figure 37).



**Figure 37** Route the heater/sensor cables

- 2 Replace the cover, vent assembly, and, on the dual-wavelength detector, the left PMT assembly. Refer to “Accessing Heaters and Ignitor” on page 58.
- 3 Carefully thread the heater/sensor cables into the electronics compartment.
- 4 Put on an ESD wrist strap. Connect the short cable as shown in Figure 6 on page 23. Connect the long cable to the auxiliary heater board (Table 11).

**Table 11** Connectors and leads

Detector type	Location	Connector
Single-wavelength	Front	A1
Single-wavelength	Back	A2
Dual-wavelength	Back	A2

## **4 Maintenance and Troubleshooting**

- 5** Replace the right-side cover to the GC electronics compartment.
- 6** Turn on the GC. Confirm that the flame is off.
- 7** Restore the operating conditions.
- 8** Wait 20 minutes for the detector to heat up, then ignite the flame.

## Replacing the Ignitor

Replacing the ignitor takes about 20 minutes. It requires you to turn off the GC, remove the vent and cover, and, on the dual-wavelength detector, the left OMT assembly. Agilent recommends that you do not touch these parts with your bare hands. For optimum performance use lint-free gloves for assembling and inserting the new parts into the emissions block assembly.

You may wish to replace the FPD ignitor for the following reasons:

- After the detector reaches operating temperatures, the FPD will not light.
- Baseline increase indicates dirt buildup.

### Materials needed

- 19256-60800 Ignitor replacement kit
- Wrenches: 1/4-inch × 5/16-inch (8710-0510)
- Torx driver: T-10 (8710-2140)
- 8650-0030 (large) or 8650-0029 (small) Nylon lint-free gloves

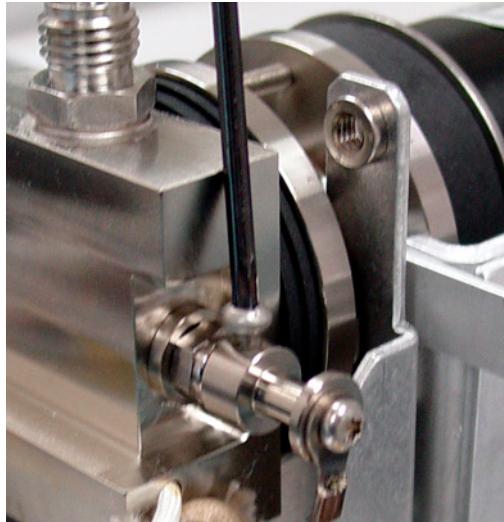
The FPD Ignitor replacement kit includes a glow plug, a spacer, and an O-ring. Do not use the copper ring that ships with the glow plug.

### Procedure

- 1 Turn off the flame.
- 2 Turn off the GC.
- 3 Remove the vent assembly, cover, and, on the dual-wavelength detector, the left PMT assembly. Refer to “Accessing Heaters and Ignitor” on page 58.

## **4 Maintenance and Troubleshooting**

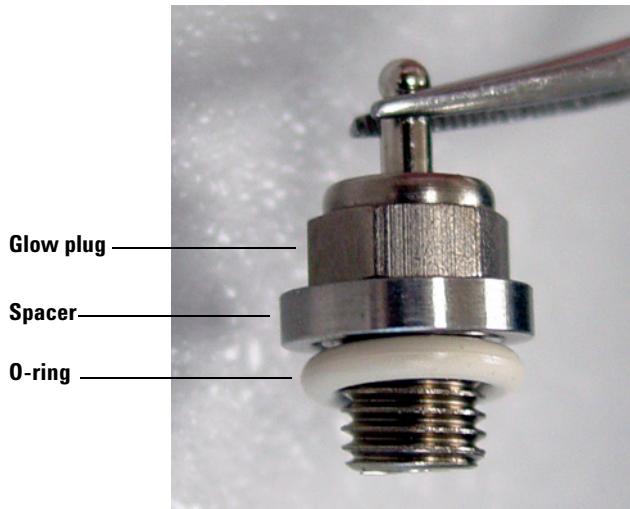
- 4** Use a Torx T-10 driver to loosen the collar screw holding the cable assembly to the ignitor (Figure 38). Remove the collar and cable assembly. Some FPDs have a version of the collar with two screws.



**Figure 38** Removing the ignitor cable

- 5** Use a wrench to loosen and remove the glow plug. Use tweezers to remove the O-ring.

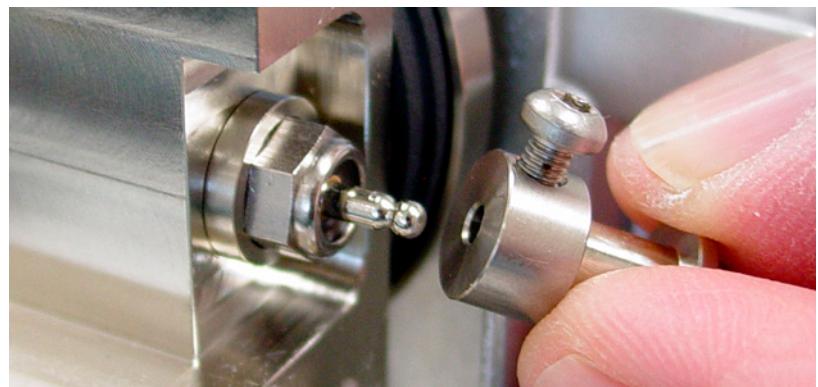
**6** Assemble the parts for the new ignitor (Figure 39).



**Figure 39** Ignitor parts assembled

**7** Insert and tighten the parts.

**8** Replace the ignitor collar and cable assembly (Figure 40). Some collars have two screws.



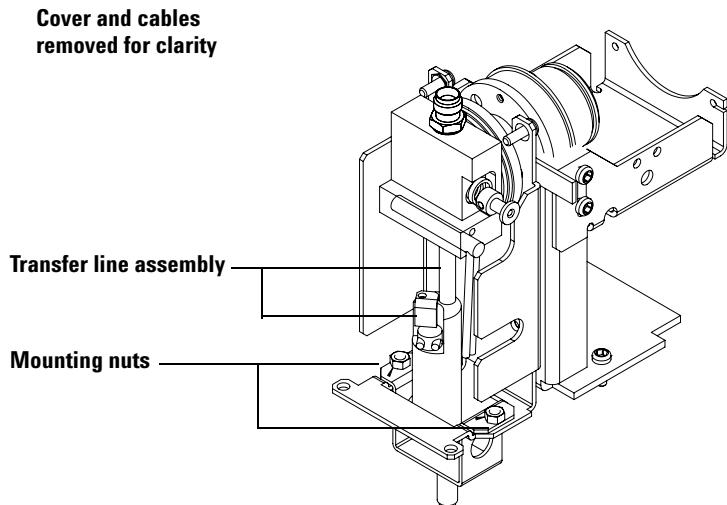
**Figure 40** Connecting the ignitor cable

## **4 Maintenance and Troubleshooting**

- 9** Replace the cover, the vent tube assembly, and, on the dual-wavelength detector, the left PMT assembly.
- 10** Turn on the GC. Confirm that the flame is off.
- 11** Restore the operating conditions.
- 12** Wait 20 minutes for the detector to heat up, then ignite the flame.

## Replacing the Inert Transfer Line

Occasionally, the inert transfer line between the column and the emissions block must be inspected, cleaned, and/or replaced. Figure 41 shows an installed inert transfer line.



**Figure 41** Left side of the detector

### Removing the transfer line

- 1 Turn off the flame.
- 2 Turn off the GC and unplug the power cord. Let the GC cool.

#### CAUTION

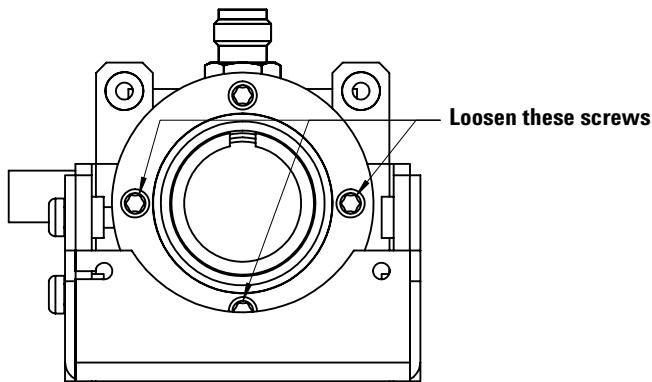
To avoid damaging the photomultiplier tube, always turn the GC or electrometer off before removing the PMT assembly.

- 3 Inside the oven, remove the column and adapter (if installed) to the FPD.
- 4 Locate the gray ignitor cable attached to the side of the detector. Trace the cable to the signal board and disconnect it there.

## 4 Maintenance and Troubleshooting

- 5 Remove the vent tubing and the sheet metal cover.  
**Single-wavelength:** two screws at the right-side top and two at the left-side bottom.  
**Dual-wavelength:** two screws at the right-side top.
- 6 Remove the PMT assemblies (release the spring, then twist and pull). It is not necessary to disconnect any cables; just place the assemblies on top of the GC and cover them to protect them from light.
- 7 Remove the two mounting nuts that hold the transfer line assembly (Figure 41).
- 8 Four screws connect the lens holder to the right face of the emissions block assembly (Figure 42). Loosen, but do not remove, the screws on the bottom and the two sides.

Similarly, loosen the screws on the left side the lens holder, if present.



**Figure 42** Lens holder and screws.

- 9 Grasp the emissions block assembly and *carefully* lift it straight up. The transfer line will come with it. Twist and pull to separate the two parts.  
If the emissions block assembly/transfer line will not lift free of the detector bracket, check for tubing or cables catching on the sheet metal.
- 10 If you will be installing a new transfer line assembly, trace the gas tubing to the manifold block on the flow module. Remove the single screw that holds the fitting. Install new O-rings.

## Reassembly

- 1** Insert the transfer line into the bottom of the emissions block assembly.
- 2** Slide the combined parts into the detector body. The semicircular plates that the three screws connect to go into the gap between the two parts of the bracket.
- 3** Install the two mounting nuts that hold the transfer line.
- 4** Check that the emissions block assembly is fully seated on the transfer line and that all of the cables and tubing are properly placed.
- 5** Tighten the screws that secure the lens holders.
- 6** Install the PMT assemblies).
- 7** Reconnect the ignitor cable.
- 8** Install the cover and the vent tube.
- 9** If necessary, connect the gas lines to the flow module.
- 10** Restore operating conditions.
- 11** Wait 20 minutes for the vent tube to heat up, then ignite the flame.

## 4 Maintenance and Troubleshooting

# Troubleshooting

## Offset or detector output too high or too low

Using a filter with flows optimized for the other filter type can cause unexpected output levels.

Monitor the FPD output. Table 12 provides examples of detector output when the filter installed in the detector and the gas flows in use do not match.

**Table 12** Filter/Flow mismatch

Gas flows optimized for	Outputs	
	With sulfur filter	With phosphorus filter
Sulfur	30 to 50	10 to 12 (low)
Phosphorus	240 to 250 (high)	30 to 50

Besides having the wrong filter for a particular set of gas flows, also consider:

- If the Lit Offset is 0.5 to 3.0, check that the flame is ON.
- If the Lit Offset is 0, check if the electrometer is turned OFF or the signal cable is disconnected.
- If the Lit Offset <30, the flame may be in the wrong position. Check detector flows, column flow, and column position.

## MDL too high

Table 13 lists typical MDL values for a checkout.

**Table 13** Typical MDL values for a checkout

	Typical range after 24 hours	Limits at installation
P filter	0.060 to 0.080	≤0.100
S filter	3.8 to 5	≤6

The MDL is dependent on the peak area and the detector noise. The relationship is: the smaller the peak area and the larger the noise measurement, the larger the MDL. If the MDL is high, check under the “High noise level” on page 77 and “Small peak area” on page 77.

## High noise level

Table 14 lists typical noise values for a checkout.

**Table 14** Typical range of values for a checkout

	Typical range after 24 hours	Limits at installation
P filter	1.6 to 3.0	$\leq 4$
S filter	2 to 4	$\leq 5$

Higher than expected noise is caused by:

- Low purity or contaminated source gases.
- Unconditioned column or ferrules.
- Dirty or contaminated inlet components, such as the liner.
- Dirty or contaminated detector parts, such as the ignitor.
- Light leak at PMT or Vent tube. Turn the flame off and check the output. If the output is above 10 or 20 pA you probably have a light leak.

## Small peak area

Table 15 lists typical peak area values for a checkout.

**Table 15** Typical peak area values for a checkout

	Typical range after 24 hours	Limits at installation
P filter	19000 to 32000	$\geq 19000$
S filter	8000 to 19000	$\geq 8000$

## 4 Maintenance and Troubleshooting

Small peak area is caused by:

- End of column extends too far into the detector. Sample not burned in flame.
- Sample lost in inlet or at column connections. Check your GC method split or splitless setpoints. Check the inlet septum, liner O-ring, and seal.
- Photo-multiplier tube defective.
- Incorrect filter or detector flows.

### Large peak width at half-height

Table 16 lists typical peak width values for a checkout.

**Table 16** Typical peak width values for a checkout

Typical range after 24 hours	
P filter	0.05 to 0.07
S filter	0.06 to 0.08

Large peak width is caused by:

- Activity in the inlet or column.
- Injection volume too large for inlet, liner, or inlet conditions.

### Detector Temperature Not Ready (6890)

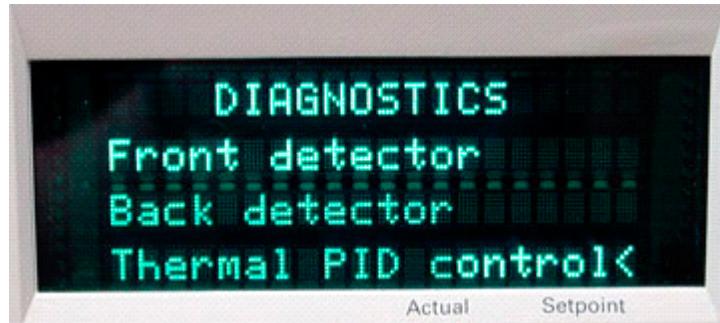
The performance of the AUX heater is affected by the placement of the PRT sensor. If it is not seated completely into the transfer line, the temperature will wander about the set point and the GC will be Not Ready. See Table 17.

**Table 17** Detector temperature range and duty cycle

PRT location	Setpoint	Duty cycle	Temperature range
Detector	200 °C	16–17%	200.02 to 199.98 °C
AUX Seated	200 °C	16–24%	200.20 to 199.8 °C
AUX not seated	200 °C	50–100%	205.00 to 195.00 °C

If the AUX temperature for the FPD is cycling or wandering, view the PID thermal diagnostic for the AUX heater by:

- 1 On the keypad, press Options.
- 2 Scroll to and select Diagnostics.
- 3 Scroll to and select Thermal PID control.



- 4 Scroll to and select AUX 1 or 2. This allows you to view the temperature range and duty cycle. Check the table above for normal ranges.



### Clipped peaks

If you have an application at the upper limit of the dynamic range (especially with sulfur), you may have to desensitize your instrument. Replace the 1000-1437 sulfur filter with filter part number 19256-80000. Then set the detector gas flows to the values used in the phosphorus checkout method. This raises the baseline but with some loss in the signal-to-noise ratio.

## 4 Maintenance and Troubleshooting

There should be no problems with either solution and the use of hydrogen. However, please note the following warning.

### WARNING

**Hydrogen gas is flammable and potentially explosive. Be sure to turn off the hydrogen gas at the source until connections are made. Also, leak test connections, lines and valves before operating or servicing the instrument.**

---

### Data lost because detector tries to reignite during a run

#### Controlling Flame Auto-Ignition

Certain environmental conditions, such as

- Strong electromagnetic fields
- Large ambient temperature swings
- Large atmospheric pressure swings

can cause an artificially low signal in the GC, incorrectly indicating that the flame has gone out. As a result, the run aborts and the GC tries to relight an already-lit flame.

You can verify that the flame is lit by holding a cold, shiny surface (such as a mirror or a wrench) over the exit tube. Condensation on the surface indicates that the flame is lit.

Reset the Lit Offset to 2.0.

### CAUTION

An attempt to relight an already-lit flame corrupts any chromatograms created and shuts down the GC.

---

### Flame extinguishes after ignition

Condensation in the vent tube because the detector was too cool when the flame was ignited.

Flame going out is caused by:

- Incorrect detector gas flows for a mix of air and hydrogen.
- Restriction to detector gas flows.

- Leak at detector EPC module.
- Condensation dripping onto flame. Prevent this by turning the flame off until detector is at temperature for 15 minutes.

## **4 Maintenance and Troubleshooting**

## **A Replacement Parts**

- Inert Transfer Line 84
- FPD Ignitor and Heat Shield Assembly 86
- FPD Lens Assembly 88
- PMT and Bracket Assemblies 90
- Covers, Flow Modules, and Electronics 92

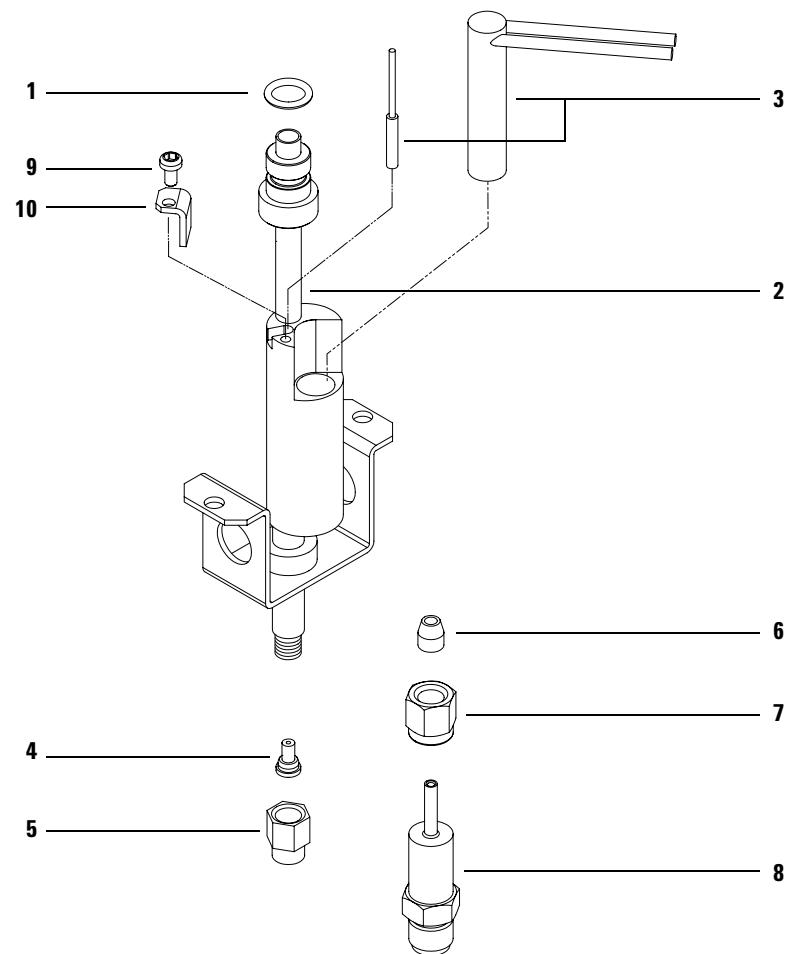


## A Replacement Parts

### Inert Transfer Line

**Table 18** Inert transfer line parts (Figure 43)

Item	Description	Part number
1	O-ring, FPD jet seal, white	0905-1608
2	Inert transfer line	19256-60555
3	Heater/Sensor assembly	G1535-60620
4	Capillary adapter seat	19256-21140
5	Capillary adapter nut	19256-21150
6	1/8-inch ferrule	0100-1332
7	1/8-inch nut	5180-4103
8	1/4-inch packed adapter	G1532-20710
9	Screw, M3 × 12 mm	0515-1084
10	Retaining clip	19256-00340



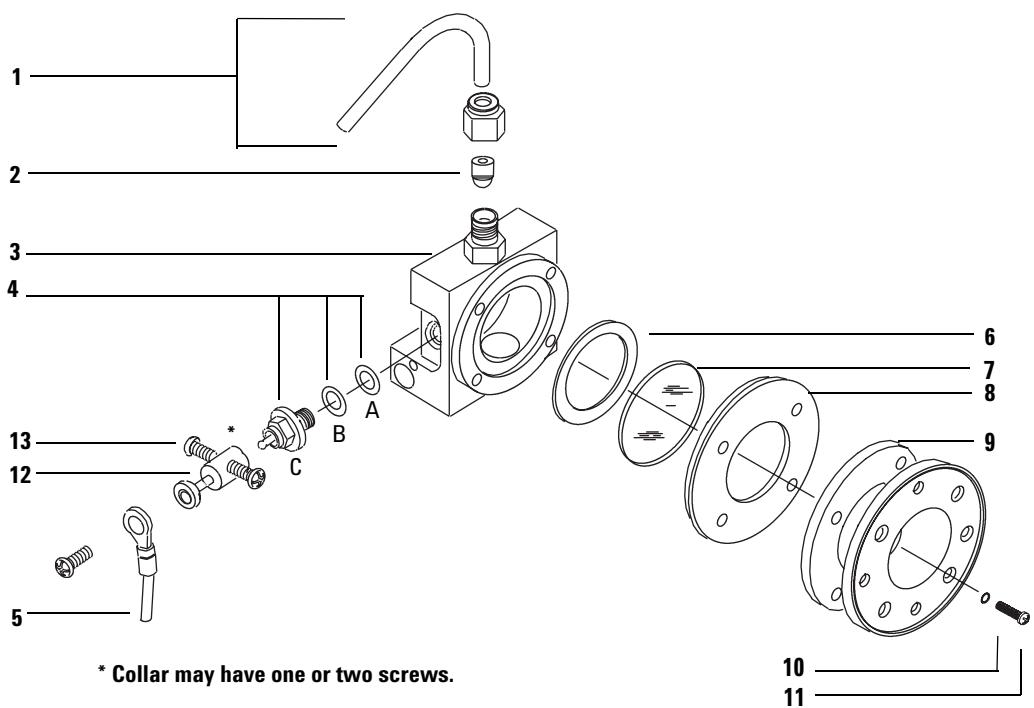
**Figure 43** Inert transfer line

## A Replacement Parts

### FPD Ignitor and Heat Shield Assembly

**Table 19** FPD ignitor and heat shield assembly parts (Figure 44)

Item	Description	Part number
1	<b>FPD tube assembly</b> Aluminum Stainless Steel	19256-60700 19256-20705
2	Vespel ferrule, 1/4-in ID	0100-1061
3	Emissions block assembly FPD, single FPD, dual (not shown)	19256-80560 19256-80600
4	<b>Ignitor replacement kit</b> A. O-ring B. Spacer C. Glow plug	19256-60800
5	Ignitor cable assembly	G1535-60600
6	Heat shield gasket, white	19256-80045
7	First heat shield window	19256-80030
8	Heat shield disk	19256-20580
9	Stainless steel coupling	19256-20550
10	Lock washer (4 required)	2190-0584
11	Screw, M3 × 12 (4 required) - T10	0515-1084
12	Collar	19256-20690
13	Screw, M3 × 66 mm, T-10	0515-0680



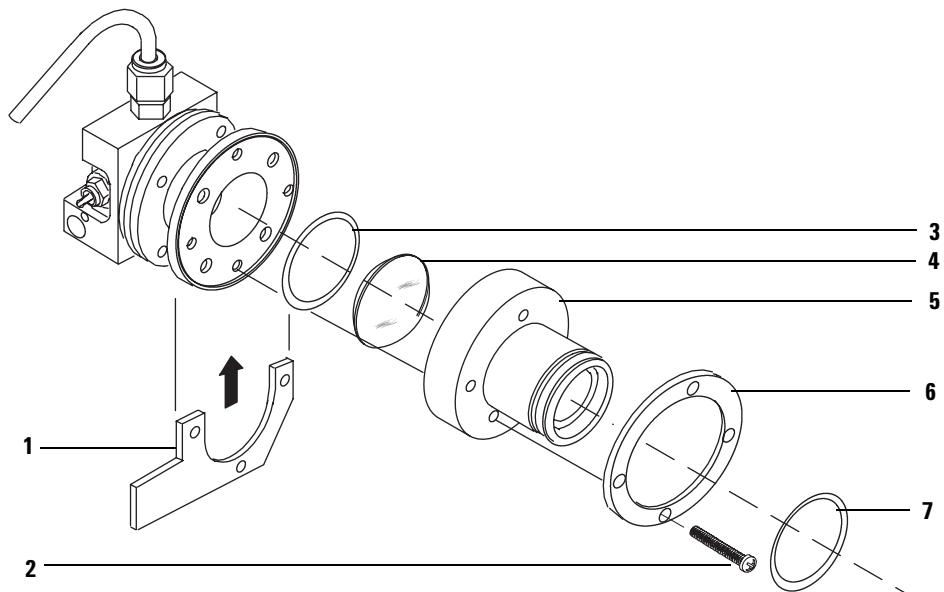
**Figure 44** FPD ignitor and heat shield assembly

## A Replacement Parts

### FPD Lens Assembly

**Table 20** FPD lens assembly parts (Figure 45)

Item	Description	Part number
1	Clamp	19256-00090
2	Screw, M3 × 25 (4 required)	0515-0683
3	Silicone O-ring, 0.926-in ID (orange)	0905-0955
4	Convex lens	1000-1438
5	Lens housing	19256-20900
6	Flange ring	19256-00200
7	O-ring, Viton, 1.239-in ID (brown), for lens housing	0905-1100



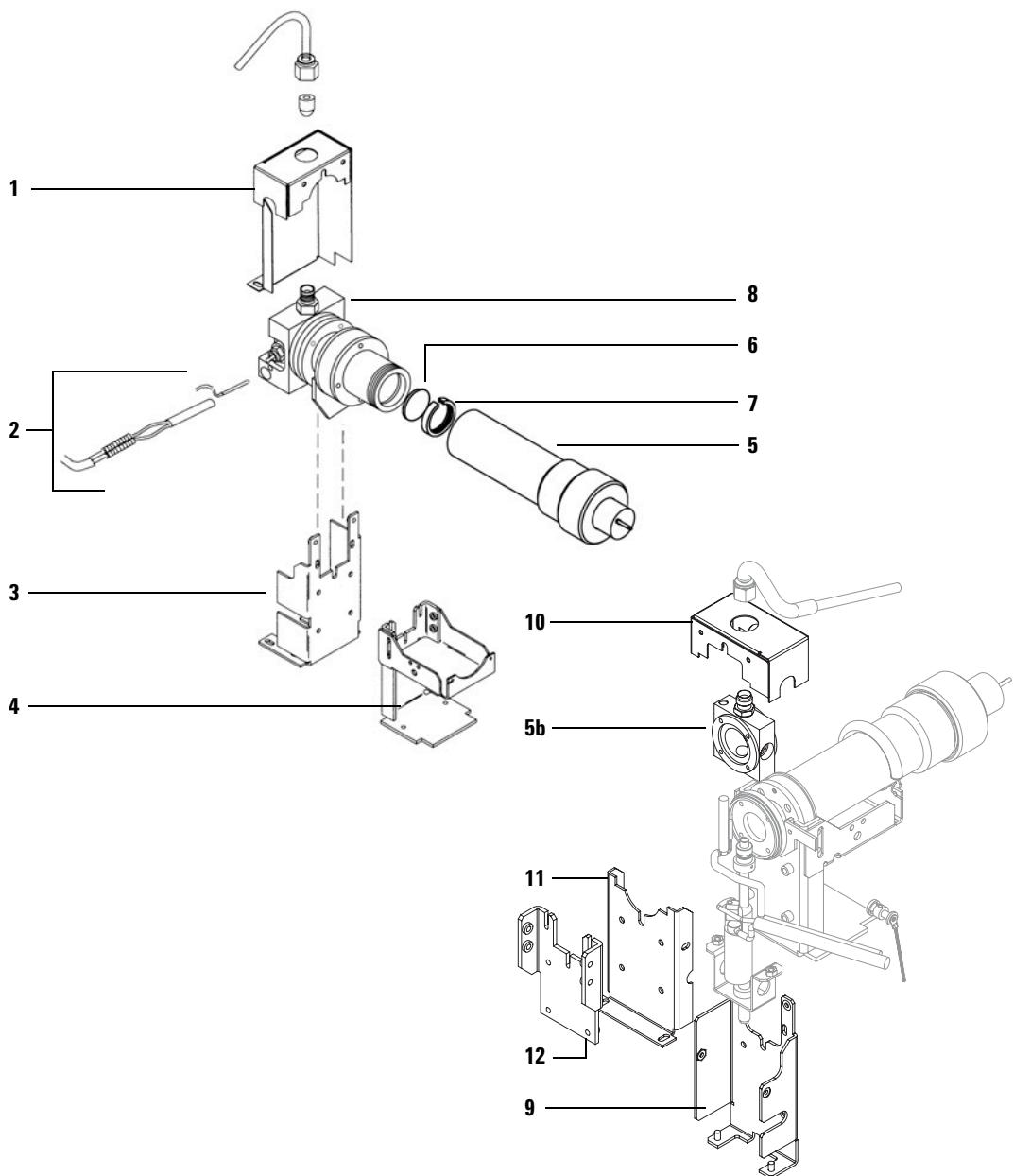
**Figure 45** FPD lens assembly

## A Replacement Parts

### PMT and Bracket Assemblies

**Table 21** PMT and Bracket Assemblies Parts (Figure 46)

Item	Description	Part number
1	Chimney back cover	G1535-80520
2	Heater/Sensor assembly	G1535-60610
3	Transfer line support bracket	19256-00320
4	Bracket/Support	G1535-00010
5	Emissions block assembly, single	19256-60650
5b	Emissions block assembly, dual	19256-60690
6	<b>Filters</b> Sulfur Phosphorus	1000-1437 19256-80010
7	Filter spacer (used only with sulfur filter)	19256-20910
8	PMT housing assembly	19256-60510
9	Dual FPD chimney front	G1535-00030
10	Dual cover assembly	G1535-80530
11	Dual FPD chimney back	19256-00330
12	Dual main bracket	G1535-00040



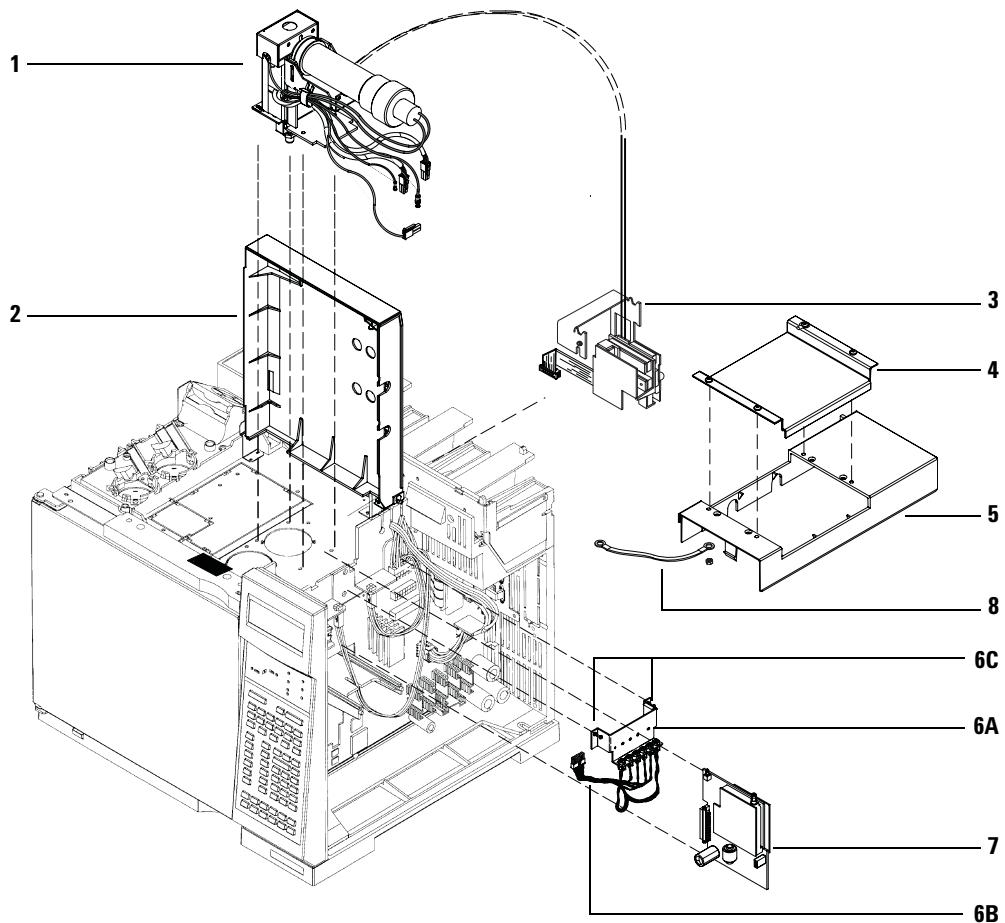
**Figure 46** PMT and bracket assemblies

## A Replacement Parts

### Covers, Flow Modules, and Electronics

**Table 22** Covers, manifolds, and electronics parts (Figure 47)

Item	Description	Part number
1	<b>FPD assembly</b> Single wavelength Dual wavelength (not shown)	G1535-61555 G1535-61565
2	<b>Top cover</b> Single wavelength Dual wavelength	G1535-80555 G1535-80560
3	Flow module kit	G1535-60720
4	Electronics cover pan	G1535-00120
5	Electronics top cover	G1535-80520
6A	Solenoid valve bracket	G1580-00070
6B	Aux zone/valve box cable	G1530-60660
6C	Screw, M4 × 12 mm (2)	1390-1023
7	Signal board	G1535-60010
8	Ground strap	19256-60730
NS	Spring to secure PMT	1460-1160
NS	FPD update kit, single wavelength	G2647A
	FPD update kit, dual wavelength	G2648A
	FPD PM kit, single wavelength	G2647-60510
	FPD PM kit, dual wavelength	G2648-60510
NS	Column measuring tool (see Figure 23 on page 50)	19256-80640



**Figure 47** Covers, manifolds, and electronics

## **A Replacement Parts**





**Agilent Technologies**

© Agilent Technologies, Inc.

Printed in USA, May 2005

G1535-90328