

# Agilent G3348B Flame Photometric Detector

For Agilent 6850 GC

**Installation and Operation** 



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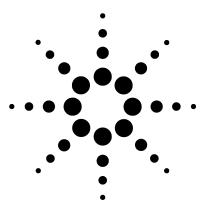
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# Installation

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The G3348 kit contains a flame photometric detector (FPD) for your Agilent 6850 Series II gas chromatograph (GC).



### 1 Installation

## **Kit Contents**

Table 1 lists the parts that come in the kit.

Description	Quantity
Screw, M4 × 12 mm long	8
Screw, M4 × 9 mm long	2
Hex nut w/lock washer	2
Disposable wrist strap	1
HOT warning label	1
Shipping kit, containing:	
Silicone tubing	1
Phosphorus filter	1
• Column nut	1
Column measuring tool	1
• Ferrule, no-hole	1
Capillary adapter seat	1
• Capillary adapter nut	1
• Ferrule, 1/8-inch, 15% graphite, 85% Vespel	1
• Nut, 1/8-inch, stainless steel	1
Packed column adapter	1
• Wrench, 3/8-inch × 7 mm	1
Detector board	1
Detector board bracket	1
Detector and flow module assembly	1
Lid top cover	1
Hinge plate	1

Table 1Parts list

#### Table 1 Parts list (continued)

Description	Quantity
Top plate (no valve box)	1
Front plate (valve box present)	1
Back plate (valve box present)	1

### NOTE

The detector is factory-assembled and tested. Do not disassemble it during installation.

## **Required Tools**

- T-20 Torx<sup>®</sup> screwdriver
- 5/16-in open-end wrench or 8-mm nut driver
- Small screwdriver
- Grounded wrist strap
- White typewriter correction fluid
- Isopropanol
- Column cutter
- Clean, lint-free gloves

#### 1 Installation

## **Overview**

## CAUTION

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

- 1 Check the contents of the kit with the list on page 8.
- **2** Prepare the GC.
- **3** Remove the current detector board.
- **4** Remove the current detector and flow module.
- **5** Install the FPD
- **6** Install the flow module.
- 7 Install the FPD detector board.
- 8 Close the lid and replace the lid top cover.
- **9** Restore the GC to operating condition.
- 10 Test the FPD (see "Checkout Procedure" on page 29 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 all gases off at their sources. Disconnect the carrier and detector gas tubing from the back panel of the instrument (see Figure 1).

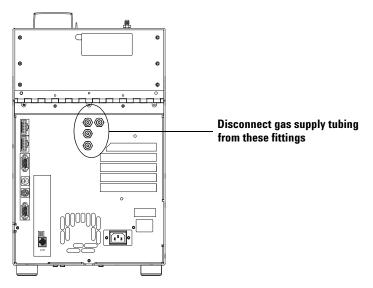
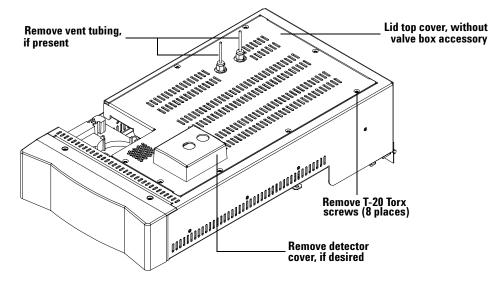


Figure 1 Disconnecting carrier and detector gas tubing

**3** Open the lid. If a column is installed, remove it and the column adapter, if present. Close the lid.



**4** Remove the lid top cover (see Figure 2).

Figure 2 Removing the lid top cover

**5** Trace the braided heater/sensor cable from the present detector to the wiring harness near the injection port cooling fan. Disconnect the cable at the connector (see Figure 3).

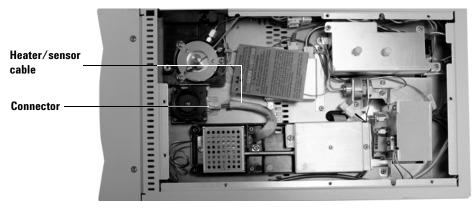


Figure 3 Disconnecting the heater/sensor cable (µECD detector shown)

**6** Open the lid. Locate the counterbalance cam in the left-rear corner under the lid. Keeping the lid open with one hand, loosen the screw on the right side of the cam and lower the stop plate. See Figure 4.

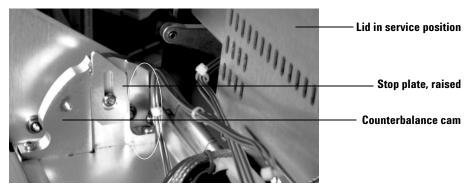


Figure 4 Opening the lid

## WARNING The lid is heavy. Always lock the lid when it is in the service position.

- 7 Raise the lid until it is stopped by the safety cable.
- **8** Raise the stop plate and tighten the screw to lock the lid in the upright service position.

## **Remove the Present Detector Board**

## CAUTION

To avoid damaging the board electronics, use the grounded wrist strap that came in the kit. Connect it to a bare metal surface of the GC.

### **Open the cover**

1 Loosen the two bottom screws on the electronics cover (Figure 5).

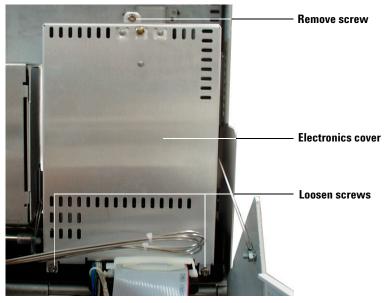


Figure 5 Removing the screws on the detector electronics cover

- **2** Remove the top screw on the cover.
- **3** Lift and remove the electronics cover.

### **Remove the present board**

 Loosen the detector board bracket. It will be either near the center of the lid (Figure 6) or at the extreme left, depending on the present detector type.

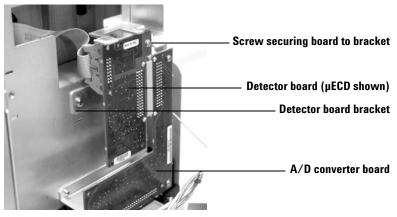


Figure 6 Loosening the detector board bracket

- 2 Unplug the detector board from the A/D converter board.
- **3** If the board bracket is near the center of the lid, as shown in Figure 6, remove it. If it is at the left side, it will be reused.

### **Disconnect wiring**

**Ribbon cables** Some detectors have a wide ribbon cable connecting the detector with the detector board. The ribbon cable connector will not pass through the grommet in the oblong hole. Use a small flat blade screwdriver to push the grommet up through the hole, then pass the cable and connector through.

Remove the cable from the grommet. Reinstall the grommet in the hole.

**FID** The FID board is small (Figure 7). Unplug the ignitor cable and the ribbon cable. Pass the ignitor cable through the hole to the top side of the lid.

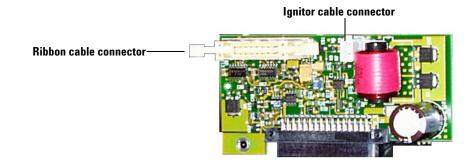


Figure 7 The FID board

**TCD** Unplug the two-wire switching valve cable at the connector (Figure 8). To release the PRT and filament wires, press down on the tabs. Pass the disconnected wires through the hole to the top side of the lid.



Figure 8 Disconnecting the TCD wires

**µECD** Remove the ribbon cable.

## **Close the lid**

- 1 Lower the stop plate on the counterbalance cam. Pull the kid forward until the cam follower rests on the counterbalance cam. Leave the stop plate in the down position.
- **2** Close the lid.

## **Remove the Present Detector and Flow Module**

## Prepare the flow module

1 On the top of the lid, loosen the connector cover plate screws and remove the plate (Figure 9).

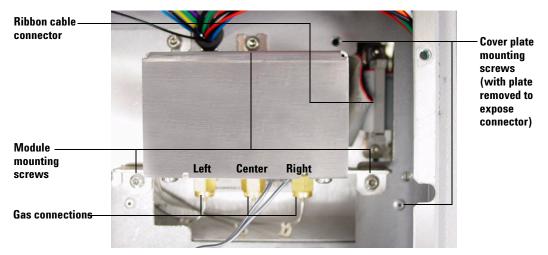


Figure 9 Mounting the flow module

- **2** Disconnect the gas fittings. The detector flow modules are similar but vary in the number of gas connections.
- **3** Trace the ribbon cable from the right side of the flow module and disconnect it.
- **4** Remove the module mounting screws.

### Prepare the detector

**FID** Remove four mounting screws (see Figure 10).

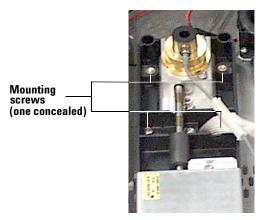


Figure 10 Removing the FID mounting screws

**TCD** Remove two mounting screws (see Figure 11).

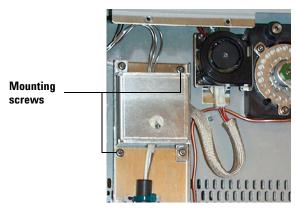
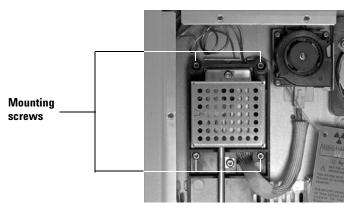


Figure 11 Removing the TCD mounting screws



**µECD** Remove four mounting screws (see Figure 12).

Figure 12 Removing the µECD mounting screws

### Remove the detector and flow module

**1** Lift the detector and flow module out of the lid.

## WARNING

The fibrous insulation material can cause irritation to the skin, eyes, and mucous membranes. Always wear gloves when working with the insulation. If the insulation is flaky/crumbly, wear protective eye wear and a respirator.

2 Remove any insulation that may be in the hole under the detector.

## **Install the FPD**

## CAUTION

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

1 Place the detector in the detector location with the column fitting extending through the insulation into the oven. The PMT housing points to the rear of the GC. The flow module is behind the housing and slightly to the left (Figure 13).

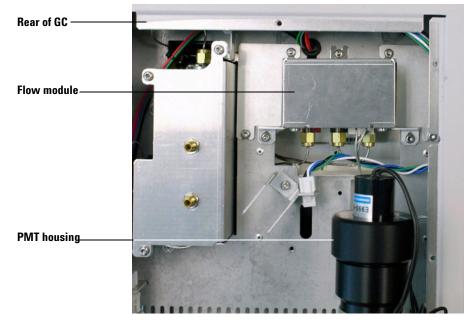


Figure 13 Installing the detector and flow module

#### 1 Installation

**2** The detector is held to the oven top by four screws, one at each corner. Insert the two screws on the rear side but do not tighten them (see Figure 14).

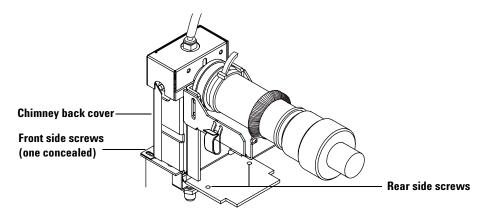


Figure 14 Installing the detector (cables removed for clarity)

- **3** Install the two screws on the front side through the sheet metal cover and the detector base. Press the chimney cover against the detector and tighten the screws.
- **4** Tighten the screws on the rear side.
- **5** Connect the braided heater/sensor cable to the heater/sensor plug under the inlet cooling fan (see Figure 3 on page 12).
- 6 There are two holes with rubber grommets under the PMT assembly:
  - Pass the gray ignitor cable on the side of the detector through the oblong hole.
  - Collect the cables from the PMT assembly into the clip on the side of the mounting bracket, then down through the large round hole.

## **Install the Flow Module**

- 1 Secure the flow module to the lid with three screws (Figure 15).
- **2** Remove the connector cover plate to the right of the module.
- **3** Plug the ribbon cable on the right end of the flow module into the connector exposed by the removed cover plate.

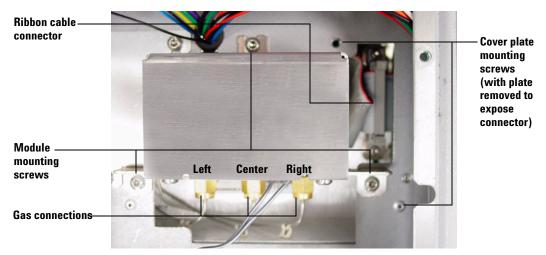


Figure 15 Mounting the flow module

**4** Connect the gas supply lines. The lines are labeled as shown in Table 2.

Table 2Gas line identification

Label on tag attached to line	Source on back panel	Connects to flow module at
н	FID H <sub>2</sub> , Not used by TCD	Left
R	Reference/Makeup Gas	Center
A	FID Air, Not Used by TCD	Right

**5** Install the connector cover plate.

## **Install the Detector Board**

### **Cable connections**

- **1** Raise the lid to the service position and lock it in place by raising the stop plate.
- **2** If needed, install the detector board bracket that came in the kit using two of the machine screws.
- **3** Connect the cables to the detector board. Figure 16 locates the connectors.



Figure 16 Detector board connector locations

#### High voltage cable

The high voltage cable is black with a chrome connector on the end (Figure 17). Press it down over the **RIGHT HI V** connector on the board until it clicks into place.



Figure 17 High voltage cable

#### **Signal cable**

The signal cable is black with a BNC connector on the end (Figure 18). Press it onto the **RIGHT PMT (DET. 1)** connector on the board and turn the knurled ring to lock it in place.



Figure 18 Signal cable

#### **Ignitor cable**

The ignitor cable is gray and ends in a 2-pin flat connector (Figure 19). Press it into either of the the ignitor connectors on the board until it locks into place. There is only one way that it can be connected.

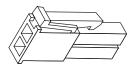
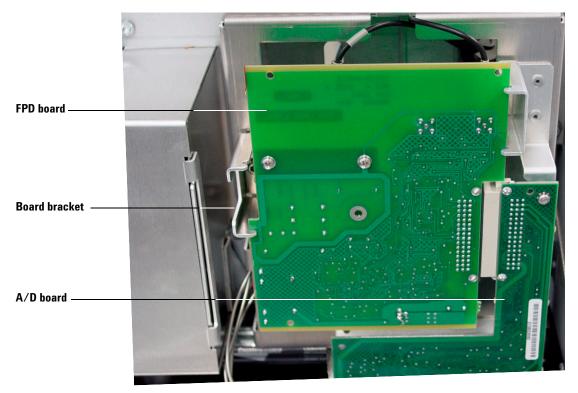


Figure 19 Ignitor connector

**4** After connecting the cables, tip the board up and plug it into the A/D converter board. Secure it with the board bracket using the bracket screw. Figure 20 shows the completed installation.

### 1 Installation





**5** Install the detector electronics cover and tighten all three screws.

## **Close the Lid and Install the Covers**

- 1 Lower the stop plate on the counterbalance cam. Pull the lid forward until the follower rests on the curved surface of the cam. Raise the stop plate behind the cam follower and tighten the screw.
- 2 Close the lid. Be sure that the folded gas lines do not interfere with it.
- **3** Install the appropriate top covers from the kit (Figure 21). Threaded studs on the hinge plate go into the top or back plate and are secured by hex nuts.

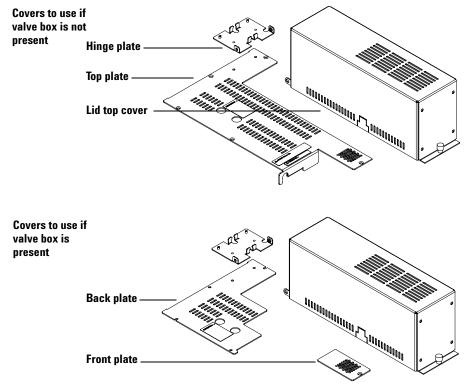
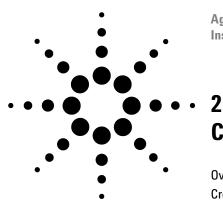


Figure 21 Installing top covers

- **4** Install the lid top cover that came in the kit.
- **5** Attach the silicone tubing to the vent tube on top of the detector. Route the tubing out the back of the lid top cover to a suitable collector or drain.

## **Restore the GC to Operating Condition**

- **1** Restore power.
- **2** Turn off the flame and electrometer.
- **3** Connect hydrogen, air, and makeup gas to the GC back panel.
- **4** Restore carrier and other gas flows.
- **5** Apply your normal operating pressures. Leak check the flow module, back panel, and column fittings.
- **6** Restore normal operating conditions. After the detector has heated for 20 minutes, turn on the electrometer and flame.



Agilent G3348 Flame Photometric Detector Installation and Operation

#### -Checkout Procedure

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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 chromatograms (but not the performance tests) can be run with just a strip-chart recorder.



#### 2 Checkout Procedure

## **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.

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

**10** When it levels off, run one sample. Interpret the results.

#### Materials needed

- Column: HP-1 Capillary 30.0 m × 0.32 mm × 0.25  $\mu$ m (part number 19091Z-413E) For Japan only, HP-5MS, 30.0 m × 0.32 mm × 0.25  $\mu$ m (part number 19091S-113E)
- 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- $\mu$ L on-column syringe for 320- $\mu$ m column (part number 5182-0836), and needle for 320  $\mu$ m 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-µL syringe (part number 5181-1267), or equivalent, and a single-taper liner deactivated without (part number 5181-3316) glass wool.

- Chromatographic-grade isooctane 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.

## **Creating Methods**

Create the test methods using these steps:

- **1** Create a phosphorus method (Table 3).
- $\label{eq:product} 2 \ \mbox{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 34).
- 4 Save the copy using a distinctive name that identifies it as a sulfur method (such as **S\_Ckout.m**).

#### **Phosphorus method**

Table 3 contains the parameters for the phosphorus method. Give the method a meaningful name that allows you to distinguish it from the sulfur method.

Parameter/Setting	Sample 5188-5953	Sample 5188-5245
Oven		
Initial temp, °C	70	70
Initial time, min	0.00 min	0.00 min
Rate 1, °C/min	25.00	10
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
Column	Agilent 19091Z-413E, HP-1, 30.0 m × 320 µm × 0.25 µm	Agilent 19091S-113E, HP-5MS 30.0 m × 320 µm × 0.50 µm
Mode	Constant pressure	Constant pressure
Pressure	25 psi	25 psi
Detector		
Temperature, °C	200 (on)	200 (on)
Hydrogen flow	75.0 mL/min (on)	75.0 mL/min (on)

 Table 3
 Phosphorus checkout method default parameters

Parameter/Setting	Sample 5188-5953	Sample 5188-5245
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
Signal		
Data rate	5 Hz	5 Hz
Туре	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
Column Comp 1	Derive from detector	Derive from detector
nlet		
Temperature, °C	200 (Split/Splitless inlet, and see below) Oven track (Cool On-Column inlet)	250, Split/splitless,
Init pressure	25.00 psi (On)	25.00 psi (On)
Gas Saver	Off	Off
Split/splitless inlet		
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
njector (if installed)		
Sample Washes	2	2
Sample Pumps	8	8
Injection Volume	1.0 μL	1.0 μL

 Table 3
 Phosphorus checkout method default parameters (continued)

Suringo Sizo		
Syringe Size	5.0 or 10 µL	5.0 or 10 µL
Prelnj Solvent A Washes	0	0
Prelnj 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
mple	part number 5188-5953	part number 5188-5245
Injection volume	1.0 μL	1.0 µL

 Table 3
 Phosphorus checkout method default parameters (continued)

Additional settings applicable to Agilent data systems are listed in Table 4. The settings available are for the GC ChemStation. Other data systems vary.

 Table 4
 Additional settings for Agilent Data Systems

Parameter/Setting	Value
Report Settings	
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 5.

Table 5	Sulfur method	parameters
14010 0	ounar mounou	paramotoro

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 6 lists the macros. They can be downloaded from www.chem.agilent.com/cag/servsup/usersoft/files/ChemStation\_Macros\_Collection.htm. Look for Data Systems, then Downloads and Utilities.

**Table 6**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.
- create and append results to logs located in the instrument subdirectory. Each line is a tab-separated list of the basic results.

Place the macros in the appropriate subdirectory (Table 7) where ChemStation can find them.

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

 Table 7
 Macro subdirectories

\* 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 22 and Figure 23 show typical online plots for sulfur and phosphorus tests using the checkout methods described in "Creating Methods" on page 32.

Figure 24 and Figure 25 show the same plots for the Japan-only sample.

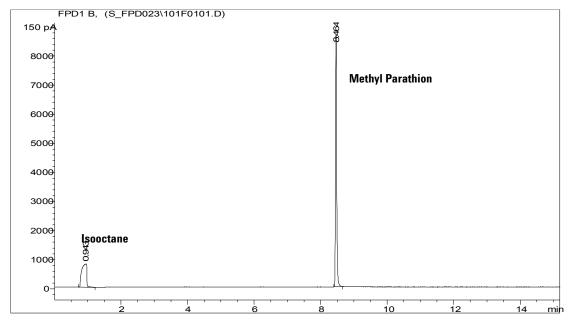


Figure 22 Typical chromatogram for sulfur tests, sample 5188-5953

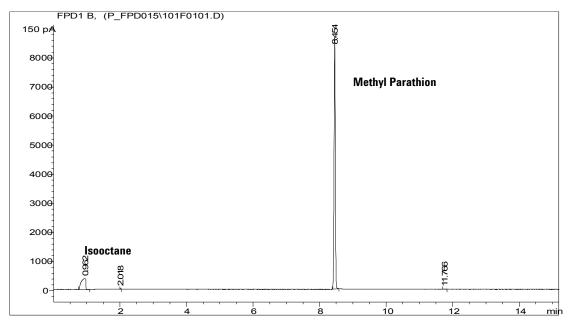


Figure 23 Typical chromatogram for phosphorus tests, sample 5188-5953

# 2 Checkout Procedure

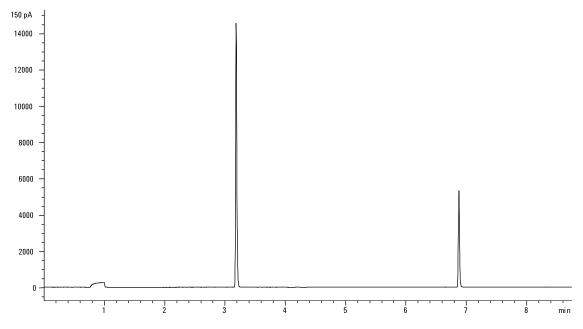


Figure 24 Typical chromatogram for sulfur tests, sample 5188-5245 (Japan only)

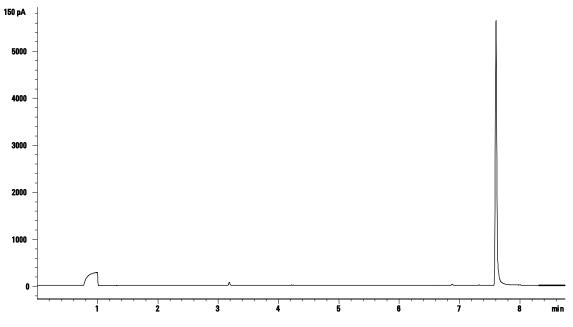


Figure 25 Typical chromatogram for phosphorus tests, sample 5188-5245 (Japan only)

# 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 contaminate 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.

# **Interpreting Results**

Compare the reported output against the values listed in Table 8 and Table 9.

# For phosphorus

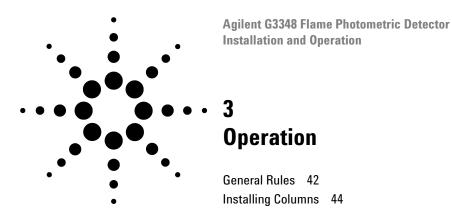
FPD P filter	Typical range after 24 hours	Limits at installation
MDL (pg/sec)	0.06 to 0.08	≤0.10
Peak area	19000 to 32000	≥19000
Signal height	5000 to 11000	
Noise	1.6 to 3.0	≤4
Half-width (min)	0.05 to 0.07	_
Signal offset	34 to 80	≤80

 Table 8
 Evaluating checkout runs

# For sulfur

FPD P filter	Typical range after 24 hours	Limits at installation
MDL (pg/sec)	3.8 to 5	<b>≤6</b>
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

 Table 9
 Evaluating checkout runs



This chapter describes **new** or **changed** operating information (at the time of this printing) that updates the information found in the 6850 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 housing or opening the emissions chamber. 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 and nitrogen makeup gases with 99.9995% purity or better. Use carbon, 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 26).

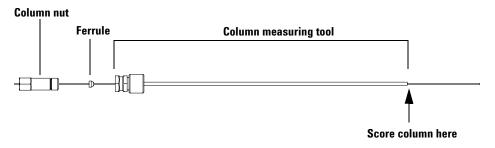


Figure 26 Cutting the column to length

- **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  $45^{\circ}$  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 27).

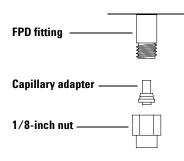


Figure 27 Capillary adapter

**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.

# Packed columns, 1/4-inch

**1** Install the adapter in the detector fitting (Figure 28). Place the nut and ferrule on the adapter, insert the adapter into the FPD fitting, and tighten the nut.

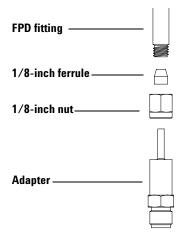
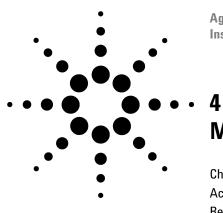


Figure 28 1/4-inch column adapter

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



Agilent G3348 Flame Photometric Detector Installation and Operation

# Maintenance and Troubleshooting

Changing the Wavelength Filter 48 Accessing Heaters and Ignitor 51 Replacing the Heater/Sensor Assemblies 55 Replacing the Ignitor 59 Replacing the Inert Transfer Line 63 Troubleshooting 66



# **Changing the Wavelength Filter**

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

# **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** Loosen the thumbscrew and raise the detector cover.

**3** Disconnect the retaining spring that holds the photomultiplier tube housing (PMT) to the bracket. With a rotating motion, pull the PMT away from the filter housing.



Figure 29 Removing the PMT housing

**4** To prevent light from damaging the PMT, immediately cap the end or place it face down.



Figure 30 Protecting the PMT

5 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**: use the wooden tip of the cotton swab to remove the filter spacer. Then remove the filter as above.

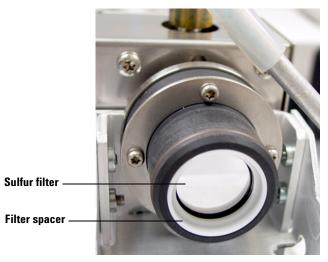


Figure 31 Sulfur filter and spacer

**6** Use the lens tissue to clean the new filter. Install it in the filter housing. If you are installing a sulfur filter, include 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.

- **7** Replace the PMT housings and secure them with the springs.
- **8** Restore the operating conditions.

# **Accessing Heaters and Ignitor**

Removing the vent and cover 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.

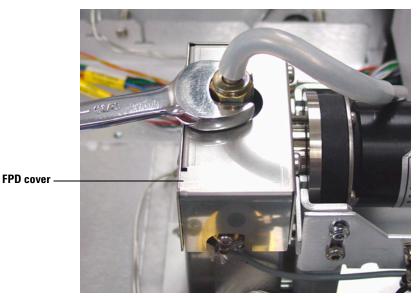
#### **Materials needed**

- 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** Loosen the thumbscrew and raise the detector cover.
- **5** Remove the drain tubing from the vent tube.



**6** Use a wrench to loosen and remove the vent tube assembly.

Figure 32 Removing the vent tube

- 7 Use a Torx T-20 driver to remove the screws securing the FPD cover. There are two screws at the bottom of the front side and two screws at the top of the rear side (Figures 33 and 34).
- **8** Lift the cover off the detector.



Figure 33 Cover screws, front side

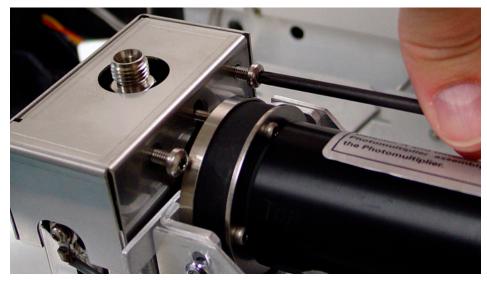


Figure 34 Cover screws, rear side

#### To reinstall the vent tube and cover

- 1 Start the 2 screws on the rear side of the cover.
- 2 Start and tighten the screws at the base on the front side.
- **3** Tighten the screws on the rear side.
- 4 Replace the vent tube assembly and connect the drain tube.
- **5** Turn on the GC. Confirm that the flame is off.
- **6** Restore the operating conditions.
- 7 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, and remove some covers.

### 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 the sensor are defective.
- The actual temperature reading on the display of the heaters is cycling more than 1 degree C.

#### **Materials needed**

- Heater/sensor assembly
- 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, drain tubing, and the detector cover. See "Accessing Heaters and Ignitor" on page 51.
- **4** Remove enough of the lid top covers to expose the detector and the braided heater/sensor cabling.
- **5** Put on an ESD wrist strap. Disconnect the heater/sensor leads from the plug near the inlet cooling fan.

#### **Transfer line**

**1** Use a Torx T-10 driver to remove the screw and retainer clip holding the lower heater. Remove the heater from the transfer line.

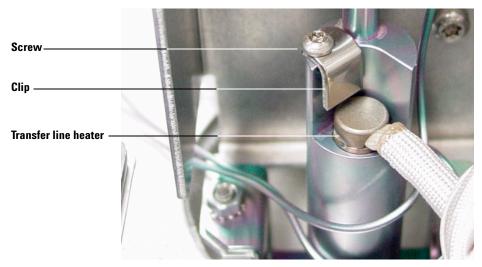


Figure 35 Replacing the transfer line heater

**2** Insert the new heater with the shorter leads into the transfer line. Position the retainer clip over the heater and install the screw.

#### **Emissions assembly**

**3** Remove the protective cap from the sensor of the heater/sensor assembly.



**4** Install the remaining heater and the sensor in the emissions block assembly. Push them in as far as they will go.

Figure 36 Install the upper heater and sensor

#### **Closing up**

1 Route the combined heater/sensor cables out the right side of the detector body, around behind it, and plug it into the connector under the inlet cooling fan (Figure 37).

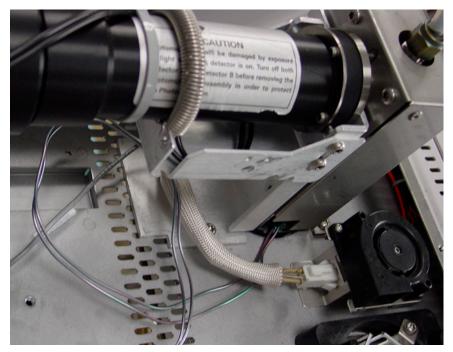


Figure 37 Routing the cables

- **2** Replace the cover and vent assembly. See "Accessing Heaters and Ignitor" on page 51.
- **3** Turn on the GC. Confirm that the flame is off.
- **4** Restore the operating conditions.
- **5** 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 and remove the vent. 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 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

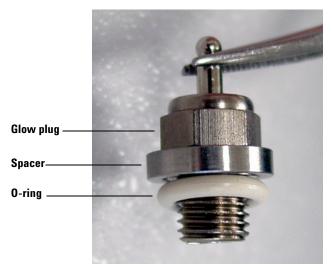
- 1 Turn off the flame.
- **2** Turn off the GC.
- **3** Remove the vent assembly and cover. Refer to "Accessing Heaters and Ignitor" on page 51.

**4** Use a Torx T-10 driver to loosen the collar screw holding the cable assembly to the ignitor. Remove the collar and cable assembly. Some FPDs have a version of the collar with 2 screws.



Figure 38 Removing the ignitor cable

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



**6** Assemble the parts for the new ignitor.

Figure 39 Ignitor parts assembled

- **7** Insert and tighten the parts.
- 8 Replace the ignitor collar and cable assembly (some collars have two screws).



Figure 40 Connecting the ignitor cable

**9** Replace the cover and the vent tube 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 assembly must be inspected, cleaned, and/or replaced. Figure 41 shows an installed inert transfer line.

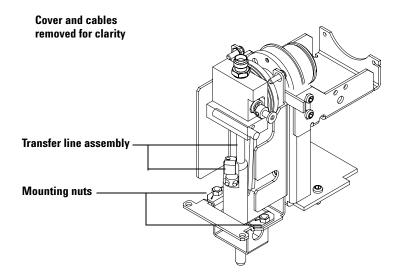


Figure 41 Left side of the detector

### **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 housing.

**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. Loosen the one (two, on some models) screw holding the collar to the glow plug. Remove the cable. See "Replacing the Ignitor" on page 59.
- **5** Remove the vent tubing and the sheet metal cover. The cover is held by two screws at the right-side top and two at the left-side bottom.
- **6** Remove the PMT housing (release the spring, then twist and pull). It is not necessary to disconnect any cables; just place the housing on top of the GC and cover it to protect it from light.
- **7** Remove the two mounting nuts that hold the transfer line assembly (Figure 41).
- 8 Three screws connect the lens holder to the rear face of the emissions assembly (Figure 42). Loosen, but do not remove, the screws on the bottom and the two sides.

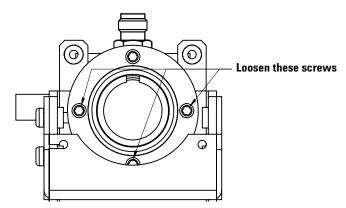


Figure 42 Lens holder and screws

**9** Grasp the emissions 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 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:

- **a** Trace the gas tubing to the manifold block on the flow module (Figure 1 on page 11). Remove the single screw that holds the fitting. Install new O-rings.
- **b** Transfer the heater/sensor assembly from the old transfer line to the new one. See "Replacing the Heater/Sensor Assemblies" on page 55.

### Reassembly

- 1 Connect the transfer line to the bottom of the emissions assembly.
- **2** Slide the combined parts onto the detector bracket. The semicircular plate that the three screws connect to goes 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 assembly is fully seated on the transfer line and that all of the cables and tubing are properly placed.
- **5** Tighten the 3 screws that secure the lens holder.
- **6** Install the PMT housing(s).
- **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.

# 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 10 provides examples of detector output when the filter installed in the detector and the gas flows in use do not match.

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

Table 10Filter/flows mismatch

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 11 lists typical MDL values for a checkout.

	Table 11	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 67 and "Small peak area" on page 67.

### **High noise level**

Table 12 lists typical noise values for a checkout.

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

**Table 12**Typical noise values for a checkout

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 13 lists typical peak area values for a checkout.

	Typical range after 24 hours	Limits at installation
P filter	19000 to 32000	≥19000
S filter	8000 to 19000	≥8000

 Table 13
 Typical peak area values for a checkout

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 14 lists 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	

**Table 14**Typical peak width values for a checkout

Large peak width is caused by:

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

### **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 phophorus checkout method. This raises the baseline but with some loss in the signal-to-noise ratio.

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 re-ignite 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.



Agilent G3348 Flame Photometric Detector Installation and Operation

# Replacement Parts

Inert Transfer Line62FPD Ignitor and Heat Shield Assembly63FPD Lens Assembly65PMT and Bracket Assemblies66Covers and Miscellaneous Parts80



# **Inert Transfer Line**

ltem	Description	Part number
1	O-ring, FPD jet seal, white	0905-1608
2	Inert transfer line	19256-60555
3	Heater/sensor assembly	G2628-60610
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	Retainer clip	19256-00340
10	Screw, M3 × 12 mm	0515-1084

 Table 15
 Inert transfer line parts (Figure 43)

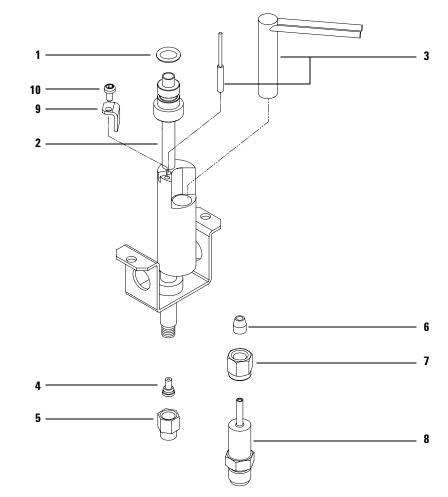


Figure 43 Inert transfer line

# FPD Ignitor and Heat Shield Assembly

ltem	Description	Part number	
1	FPD tube assembly		
	Aluminum	19256-60700	
	Stainless steel	19256-20705	
2	Vespel ferrule, 1/4-inch ID	0100-1061	
3	Block weldment	19256-80560	
4	Ignitor replacement kit	19256-60800	
	A. O-ring		
	B. Spacer		
	C. Glow plug		
5	Ignitor cable assembly	G1535-60600	
6	Heat shield gasket, white	19256-80045	
7	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 mm (4 required), T-10	0515-1084	
12	Collar	19256-20690	
13	Screw, M3 × 6 mm	0515-0680	

**Table 16**FPD ignitor and heat shield assembly parts (Figure 44)

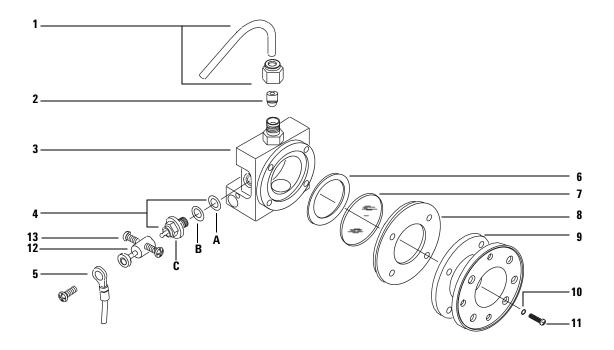
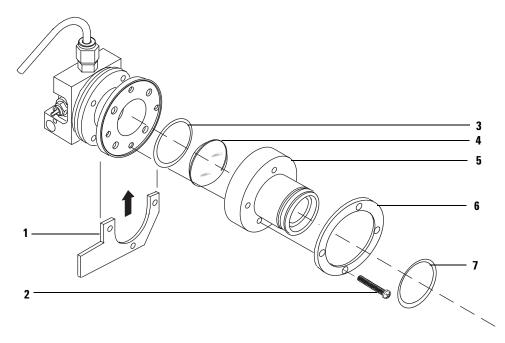


Figure 44 FPD ignitor and heat shield assembly

# **FPD Lens Assembly**

ltem	Description	Part number
1	Clamp	19256-00090
2	Screw, M3 × 25 (4 required) - T10	0515-0683
3	Silicone O-ring .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)	0905-1100

 Table 17
 FPD lens assembly parts (Figure 45)





## **PMT and Bracket Assemblies**

ltem	Description	Part number
1	Chimney back cover	G1535-80520
2	Heater/sensor assembly	G2628-60610
3	Transfer line support bracket	19256-00320
4	Bracket/support	G1535-00010
5	PMT housing assembly	19256-60510
6	Filters	
	Sulfur	1000-1437
	Phosphorus	19256-80010
7	Spacer, used with sulfur filter	19256-20910
8	Emissions block assembly, single	19256-60650

 Table 18
 PMT and bracket assemblies parts (Figure 46)

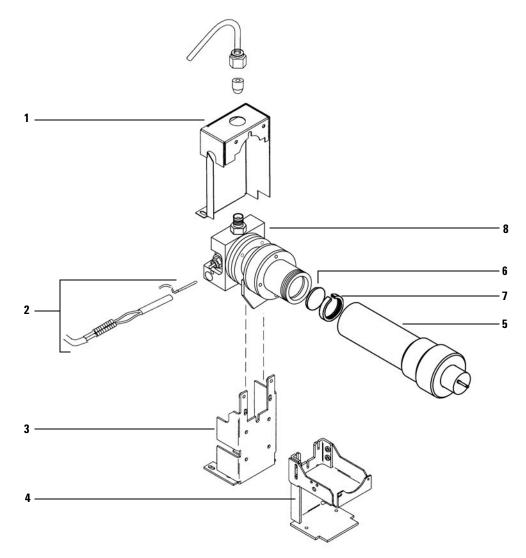


Figure 46 PMT and bracket assemblies

### **Covers and Miscellaneous Parts**

ltem	Description	Part number
1	FPD cover	G2649-00010
2	FPD hinge plate	G2649-00020
3	FPD top plate	G2649-00030
4	FPD front plate	G2649-00040
5	FPD back plate	G2649-00050
NS	Detector board	G2628-60010
NS	Nut, hex, 7 mm	0535-0043
NS	FPD preventive maintenance kit, single	G2647-60510
NS	Column measuring tool	19256-80640
NS	FPD flow module kit <sup>*</sup>	G1535-60720
NS	Screw, M4 × 12 mm (3 required)	0515-2496
NS	Nut, hex, 5/16-inch (3 required)	2950-0232
NS	Flow module cover, front	G2630-00410
NS	Flow module cover, back	G2630-00420

 Table 19
 Covers and miscellaneous parts (Figure 47)

\* The flow module must be enclosed in the front and back covers, listed below. They are not included in the kit. They may be salvaged from the old flow module or purchased using the next 4 items

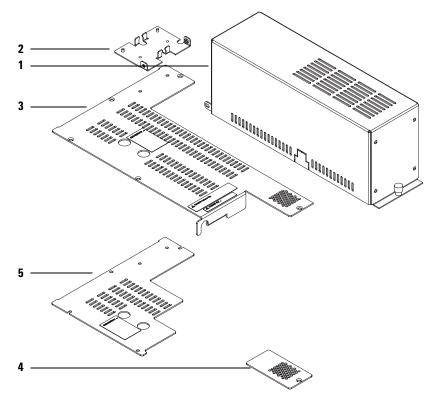


Figure 47 Covers

#### A Replacement Parts



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