Operating Manual
Volume 3. Detectors

Agilent 6890 Series Gas Chromatograph

Part No. G1530-90467

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Printed in USA

Safety Information

The 6890 Gas Chromatograph meets the following IEC (International Electrotechnical Commission) classifications: Safety Class 1, Transient Overvoltage Category II, and Pollution Degree 2.

This unit has been designed and tested in accordance with recognized safety standards and designed for use indoors. If the instrument is used in a manner not specified by the manufacturer, the protection provided by the instrument may be impaired. Whenever the safety protection of the 6890 has been compromised, disconnect the unit from all power sources and secure the unit against unintended operation.

Refer servicing to qualified service personnel. Substituting parts or performing any unauthorized modification to the instrument may result in a safety hazard. Disconnect the AC power cord before removing covers. The customer should not attempt to replace the battery or fuses in this instrument. The battery contained in this instrument is recyclable.

Safety Symbols

Warnings in the manual or on the instrument must be observed during all phases of operation, service, and repair of this instrument. Failure to comply with these precautions violates safety standards of design and the intended use of the instrument. Agilent Technologies assumes no liability for the customer's failure to comply with these requirements.

WARNING

A warning calls attention to a condition or possible situation that could cause injury to the user.

CAUTION

A caution calls attention to a condition or possible situation that could damage or destroy the product or the user's work.

See accompanying instructions for more information.

Indicates a hot surface.

Indicates hazardous voltages.

Indicates earth (ground) terminal.

Indicates radio-active hazard.

Indicates explosion hazard.

Electromagnetic Compatibility

This device complies with the requirements of CISPR 11. Operation is subject to the following two conditions:

1. This device may not cause harmful interference.

2. This device must accept any interference received, including interference that may cause undesired operation.

If this equipment does cause harmful interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try one or more of the following measures:

1. Relocate the radio or television antenna.

2. Move the device away from the radio or television.

3. Plug the device into a different electrical outlet, so that the device and the radio or television are on separate electrical circuits.

4. Make sure that all peripheral devices are also certified.

5. Make sure that appropriate cables are used to connect the device to peripheral equipment.

6. Consult your equipment dealer, Agilent Technologies, or an experienced technician for assistance.

7. Changes or modifications not expressly approved by Agilent Technologies could void the user's authority to operate the equipment.

Sound Emission Certification for Federal Republic of Germany

Sound pressure Lp
< 65 dB(A)
During normal operation
At the operator position
According to ISO 7779 (Type Test)

When operating the 6890 with cryo valve option, the sound pressure 74.6 dB(A) during cryo valve operation for short burst pulses.

Schallemission

Schalldruckpegel LP < 65 dB(A)
Am Arbeitsplatz
Normaler Betrieb
Nach DIN 45635 T. 19
(Typprüfung)

Bei Betrieb des 6890 mit Cryo Ventil Option treten beim Oeffnen des Ventils impulsfoermig Schalldrucke Lp bis ca. 74.6 dB(A) auf.

Agilent Technologies, Inc.
2850 Centerville Road
Wilmington, DE 19808-1610

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Part 1. General Information

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Using Detectors

Chapter 1
Using Detectors

The 6890 Series gas chromatograph (the GC) has several detector systems available. Others will be added in the future. See your Agilent sales representative for the latest information.

Name	Sensitivity	Responds to	Comments
Thermal conductivity, TCD	Medium	Everything except the carrier gas	The "Universal Detector" for everything
Flame ionization, FID	High	Almost all organic compounds	The "Universal Detector" for organics
Electron capture, ECD and μ-ECD	Very high	Limited range of compounds, mostly halocarbons	Used for trace level pesticide and herbicide analysis
Nitrogen-phosphorus, NPD	Very high	Compounds with nitrogen or phosphorus	Used in pharmaceutical and environmental analysis
Flame photometric, FPD	High	Compounds with sulfur or phosphorus	Used in environmental and bioscience analysis

Using hydrogen

WARNING When using hydrogen (H_2) as the carrier gas or fuel gas, be aware that hydrogen (H_2) gas can flow into the oven and create an explosion hazard. Therefore, be sure that the supply is off until all connections are made, and ensure that the inlet and detector column fittings are either connected to a column or capped at all times when hydrogen (H_2) gas is supplied to the instrument.

WARNING Hydrogen (H_2) is flammable. Leaks, when confined in an enclosed space, may create a fire or explosion hazard. In any application using hydrogen (H_2), leak test all connections, lines, and valves before operating the instrument. Always turn off the hydrogen (H_2) supply at its source before working on the instrument.

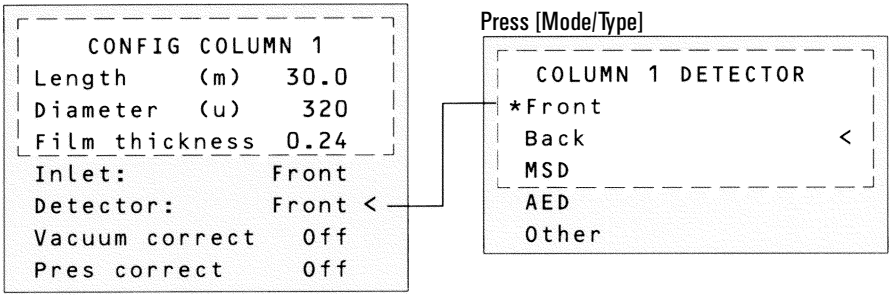
Procedure: Setting up detector control tables

You must be familiar with this set of control tables to operate your detector. Follow these three steps when setting up all types of detectors.

1. Check your column configuration. (This is normally done when you set up your inlet, but it does not hurt to check this information again.)

   - You must tell the instrument which detector your column is connected to, front or back. If you have only one detector, it is best to have only one column configured to it—unless you actually do have two columns attached to that detector.
   - If you have an EPC inlet and detector and are using a capillary column, you must enter the column length and diameter if you want to have a choice of makeup gas flow modes. This is called *column defined*. If you do not enter these values, it is called *column not defined*, and your control choices are limited.

1. Press [Config] [Col 1] or [Config] [Col 2]:



2. Scroll to `Detector:` Press [Front] or [Back] or Press [Mode/Type] and choose `Front` or `Back`
3. Enter column dimensions, if necessary.

2. Check your detector configuration (makeup gas type).

   The main reason for doing this is to verify that the makeup gas entered (or makeup and anode gas on the ECD, reference and makeup on the TCD) is the same as the gas plumbed to your detector.

   This is important because:

   - When the actual and configured gas types are different for an EPC detector, the calculated flow rate is not correct and the flow rate stability is affected.
   - The electronics for some detectors change depending on the gas type configuration. The detector does not operate properly when the actual and configured gas types are different.
   - Good laboratory practice. Keep a record of the gas used.

   Most of the detectors have other configurable items. These will be described later in this section.

3. Set up your detector control table. The following is a brief description of each line item for the FID. If you have EPC, flow setpoints (right number) and actual values (left number) are displayed. NonEPC detectors have on/off control.

Press [Front Det] or [Back Det].

EPC (column not defined)

```
    FRONT DET (FID)
Temp        250   250 <
H2 flow    40.0  40.0
Air flow  450.0 450.0
Mode:   Const makeup
Mkup (N2) 50.0  50.0
Flame             On
Output            15
```

NonEPC

```
    FRONT DET (FID)
Temp        250   250 <
H2 flow           On
Air flow          On
Mkup (N2)         On
Flame             On
Output            15
```

`FRONT DET (FID)` The title indicates the detector position and the type of detector installed.

`Temp` This is where you set the temperature. The control is identical on EPC and nonEPC detectors and includes setpoint (right number) and actual (left number) values.

`H2 flow, Air flow` These are the detector gases for the FID.

`Mkup (N2)` This is where you set your makeup gas flows. The gas type is displayed in parentheses. The lines of the display vary depending on your instrument and the way you have configured it.

`Flame` This is the on/off control for the FID—also called the Detector Control line. Each detector has its own type of on/off control.

`Output` This is the actual detector output value. You cannot enter a setpoint here.

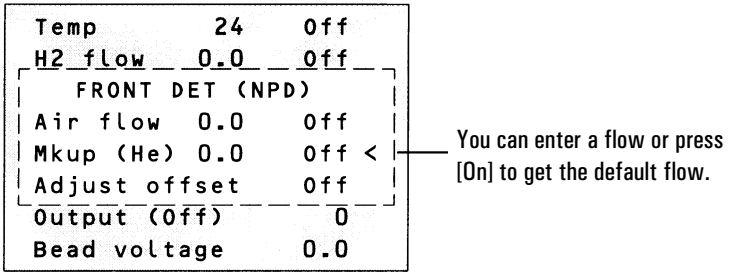
Makeup gas flow

Makeup gas enters the detector close to the end of the column. Its purpose is to speed the peaks through the detector—especially with capillary columns—so that the peak separation achieved by the column is not lost through remixing in the detector.

Makeup gas and EPC detectors

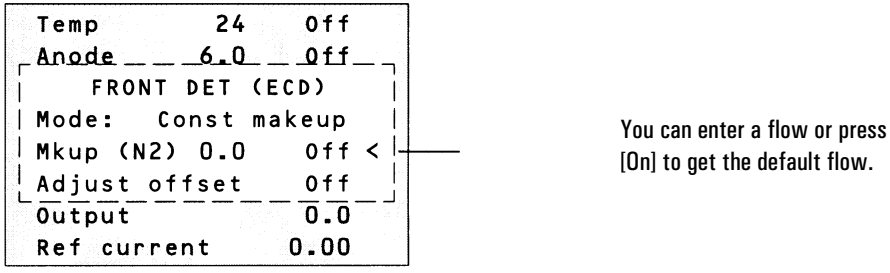
The makeup gas line of your detector control table changes depending on your instrument configuration.

If you have a nonEPC inlet or an EPC inlet with the *column not defined*, the makeup flow is constant. The control table for the EPC detector looks like this:

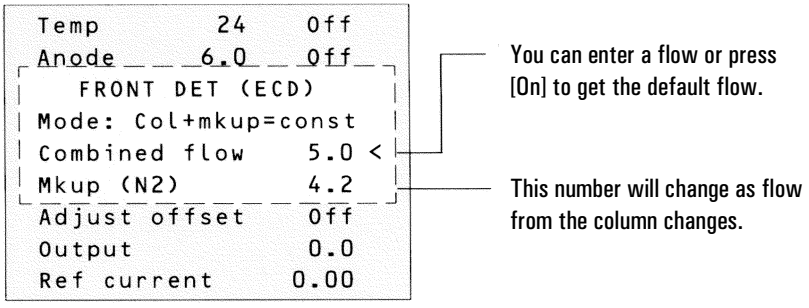


If you have an EPC inlet configured to your detector and you are operating with *column defined*, you have a choice of two makeup gas modes.

The `Const makeup` mode provides a constant flow of makeup gas to the detector. If you choose it, your control table looks like this:

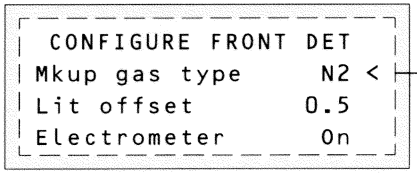


The `Col+mkup=const` mode provides a variable flow of makeup gas to the detector. As column flow increases or decreases, the makeup flow changes to provide a constant combined flow to the detector. If you choose this option, enter a value under `Combined flow.` The `Combined flow` line always displays the same value, while the `Mkup` line of the control table changes as the actual makeup flow changes.



Procedure: Defining the makeup gas

1. Press [Config] [Front Det] or [Config] [Back Det]:

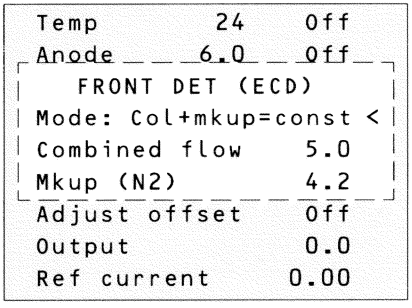


2. Scroll to `Mkup gas type` and press [Mode/Type].

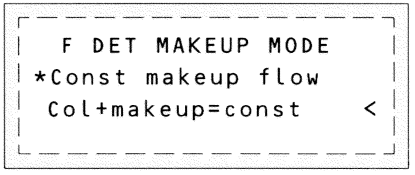
3. Scroll to the correct gas and press [Enter].

Procedure: Changing makeup gas flow mode

1. Scroll to `Mode:`



2. Press [Mode/Type].

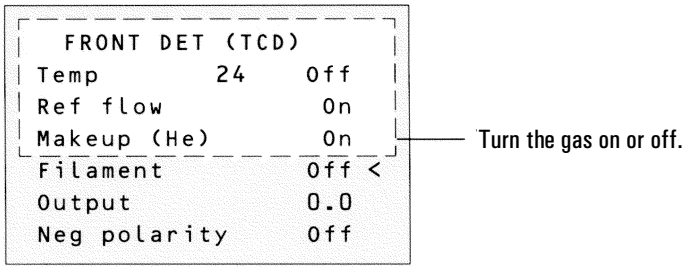


3. Choose a flow mode and press [Enter].

Makeup gas and nonEPC detector

If you have a *nonEPC detector*, your makeup gas must be measured and adjusted manually from a pressure regulator located in the pneumatics carrier. The control table only displays one flow mode — On/Off.

NonEPC detector makeup gas flow mode



Maximum flow rates

The maximum flow rates that can be set with EPC detectors are:

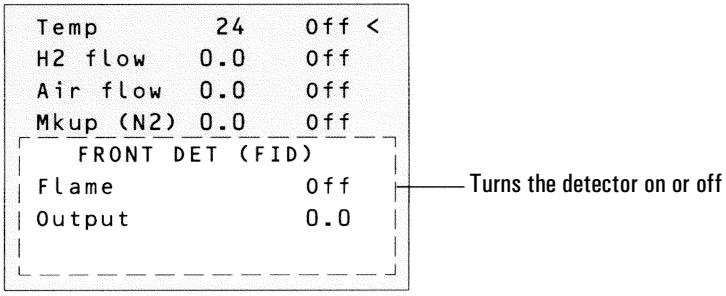
Detector and gas	Maximum flow rate, mL/min
Flame ionization	
Hydrogen	100
Air	800
Makeup (nitrogen, helium, argon)	100
Thermal conductivity	
Nitrogen	reference 100; makeup 10
Helium	reference 100; makeup 12
Hydrogen	reference 100; makeup 18
Argon	reference 100; makeup 10
Electron capture	
Nitrogen	anode purge 12; makeup 200
Argon/methane	anode purge 12; makeup 200
Nitrogen-phosphorus	
Hydrogen	30
Air	200
Makeup (nitrogen, helium, argon)	100
Flame photometric	
Hydrogen	250
Air	200
Makeup (nitrogen, helium, argon)	130

[Det Control] shortcut key

This is another way to open a Detector control table. Press [Front Det] [Det Control] or [Back Det] [Det Control] to open a Detector control table. If you have only one detector, [Det Control] opens that table.

When you use [Det Control], your table opens at the On/Off control for your detector—FID and FPD, `Flame`, TCD `Filament`, NPD and ECD, `Adjust offset`.

Press [Det Control]



2

The Flame Ionization Detector

Chapter 2
The Flame Ionization Detector

Part 1. General Information

The flame ionization detector passes sample and carrier gas from the column through a hydrogen-air flame. The hydrogen-air flame alone creates few ions, but when an organic compound is burned there is an increase in ions produced. A polarizing voltage attracts these ions to a collector located near the flame. The current produced is proportional to the amount of sample being burned. This current is sensed by an electrometer, converted to digital form, and sent to an output device.

FID pneumatics

Figure 1 and Figure 2 illustrate the pneumatics design for the FID with and without EPC.

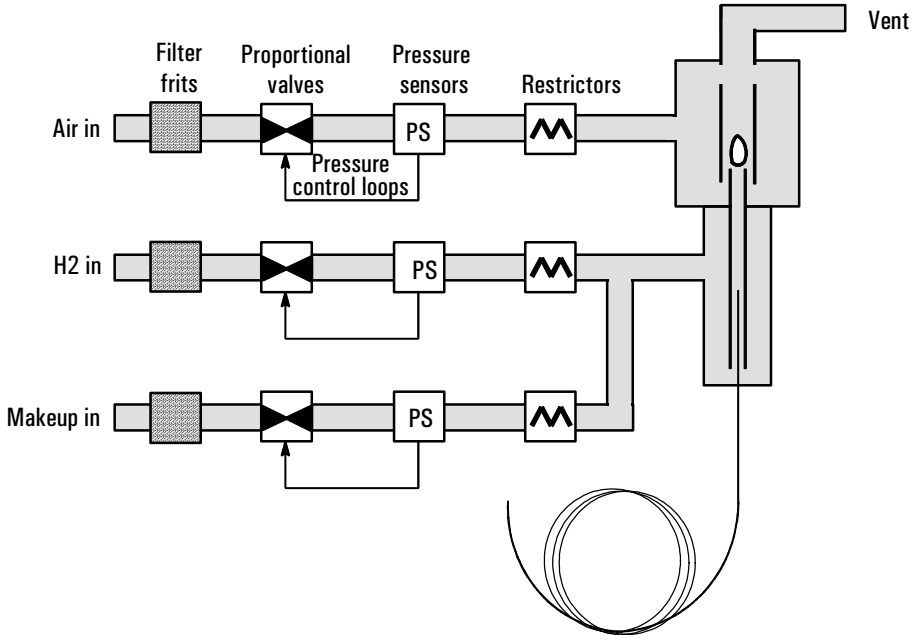


Figure 1. Schematic of a flame ionization detector — EPC

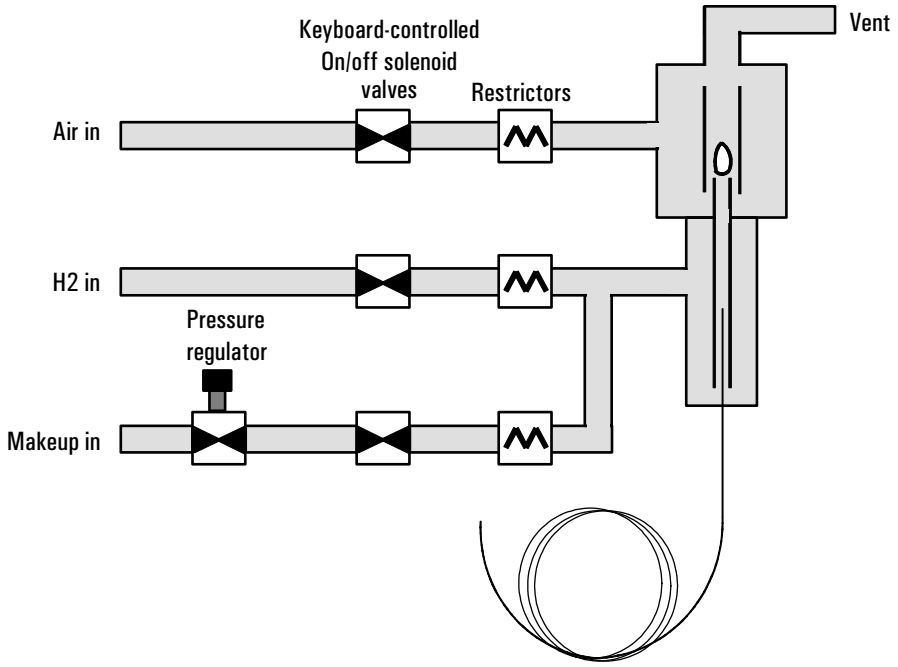


Figure 2. Schematic of a flame ionization detector—NonEPC

Special considerations

Conditions that prevent the detector from operating

- Temperature set below 150°C
- Air or hydrogen flow set at `Off` or set at 0.0
- Ignition failure

Detector shutdown

If a critical detector gas is shut down due to a pneumatics or ignition failure, your detector shuts down. This turns off everything except the detector temperature and makeup gas flow.

Jets

There are two types of FID available. The *capillary optimized* FID is only used with capillary columns, and the *adaptable* FID fits packed columns and can be adapted to fit capillary columns.

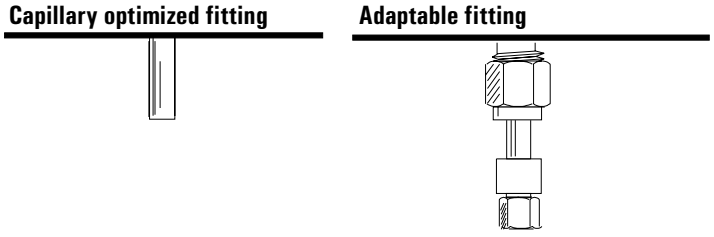


Table 1. Jets for the Capillary-Optimized FID

Jet type	Part no.	Jet tip id
Capillary	G1531-80560	0.29 mm (0.011-inch)
High-temperature *(use with simulated distillation)*	G1531-80620	0.47 mm (0.018-inch)

Table 2. Jets for the Adaptable FID

Jet type	Part no.	Jet tip id
Capillary	19244-80560	0.29 mm (0.011-inch)
Packed	18710-20119	0.47 mm (0.018-inch)
Packed wide-bore *(use with high-bleed applications)*	18789-80070	0.030-inch
High-temperature *(use with simulated distillation)*	19244-80620	0.47 mm (0.018-inch)

Your detector is shipped with a capillary column jet. If you are doing simulated distillation or high-temperature runs, you must change the jet. Instructions appear later in this chapter.

Automatic reignition—Lit offset

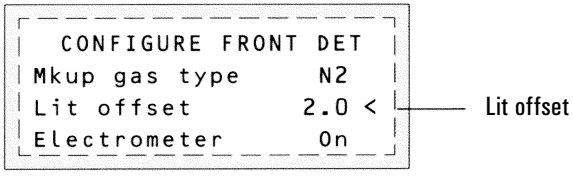
`Lit offset` is the expected difference between the FID output with the flame lit and the output with the flame off. If the output falls below this value, the FID will attempt to reignite twice. If the output does not increase by at least this value, the detector shuts down all functions except temperature and makeup gas flow.

The default setting for `Lit offset` is 2.0 picoamps. This is a good working value for all but very clean gases and systems. You may want to change this setpoint if:

- Your detector is attempting to reignite when the flame is still on, thus producing a shutdown.
- Your detector is not trying to reignite when the flame is out.

Procedure: Changing the auto-reignite setpoint

1. [Press [Config][Front Det] or [Config][Back Det].



2. Scroll to `Lit offset` and enter a number.
The default is 2.0 pA.
Enter 0 to disable the automatic reignite function.
The setpoint range is 0 to 99.9 pA.

Electrometer

The Configure Detector control table contains an `On/Off` setpoint for the `Electrometer`. You do not need to turn the electrometer on and off when operating your FID. The only time you need to turn off the electrometer is when cleaning the detector.

Caution Do not turn off the electrometer during a run. It will cancel detector `Output`.

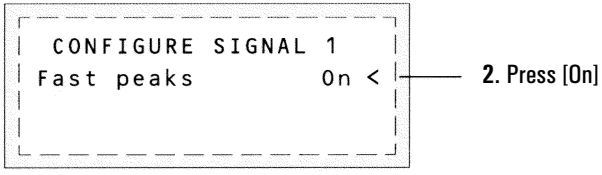
Data rates

Analog output for the FID can be presented at either of two speeds. The faster speed allows minimum peak widths of 0.004 minutes, while the standard speed allows peak widths of 0.01 minutes.

Procedure: Using fast peaks

If you are using the *fast peaks* feature, your integrator must be fast enough to process the data coming from the GC. It is recommended that your integrator bandwidth be at least 15 Hz. To use fast peaks:

1. Press [Config][Signal 1] or [Config][Signal 2]



Digital output to the ChemStation is available at eleven speeds ranging from 0.1 Hz to 200 Hz, capable of handling peaks from 0.001 to 2 minutes wide. Output to an INET integrator is available at 20 Hz. Consult “Signal Handling” in the *General Information* volume for a discussion of the different rates.

The fast peaks feature does not apply to digital output.

Part 2. Operating the FID

Use the information in Table 3 when selecting temperatures and flows. Choose a minimum source pressure from Figure 3. If you have an EPC detector, you must add 10 psi (69 kPa) to the source pressure on the chart.

Table 3. Recommended Temperature and Flow Rates—FID

Gas	Flow range (mL/min)	Suggested flow (mL/min)
Carrier gas *(hydrogen, helium, nitrogen)*		
Packed columns	10 to 60	
Capillary columns	1 to 5	
Detector gases		
Hydrogen	24 to 60*	40
Air	200 to 600*	450
Column plus capillary makeup *Recommended: nitrogen* *Alternate: helium*	10 to 60	50
Detector temperature		
< 150°C, flame will not light, prevents condensation damage		
Detector temperature should be approximately 20°C greater than highest oven ramp temperature depending on the column type.		
Lit offset [Config][Front Det] or [Config][Back Det]		
If the detector output (when the flame is on) minus the detector output (when the flame is off) falls below this value, the FID attempts to reignite twice. If output does not increase by at least this value, the detector shuts down.		
2.0 pA is the recommended setting.		
0.0 pA disables the autoreignite function.		

*The hydrogen-to-air ratio should be between 8% and 12% to keep the flame lit.

Gas pressures

NonEPC detector—choose a flow, find a starting source pressure.
EPC detector—choose a flow, find a pressure. Set source pressure 10 psi higher.

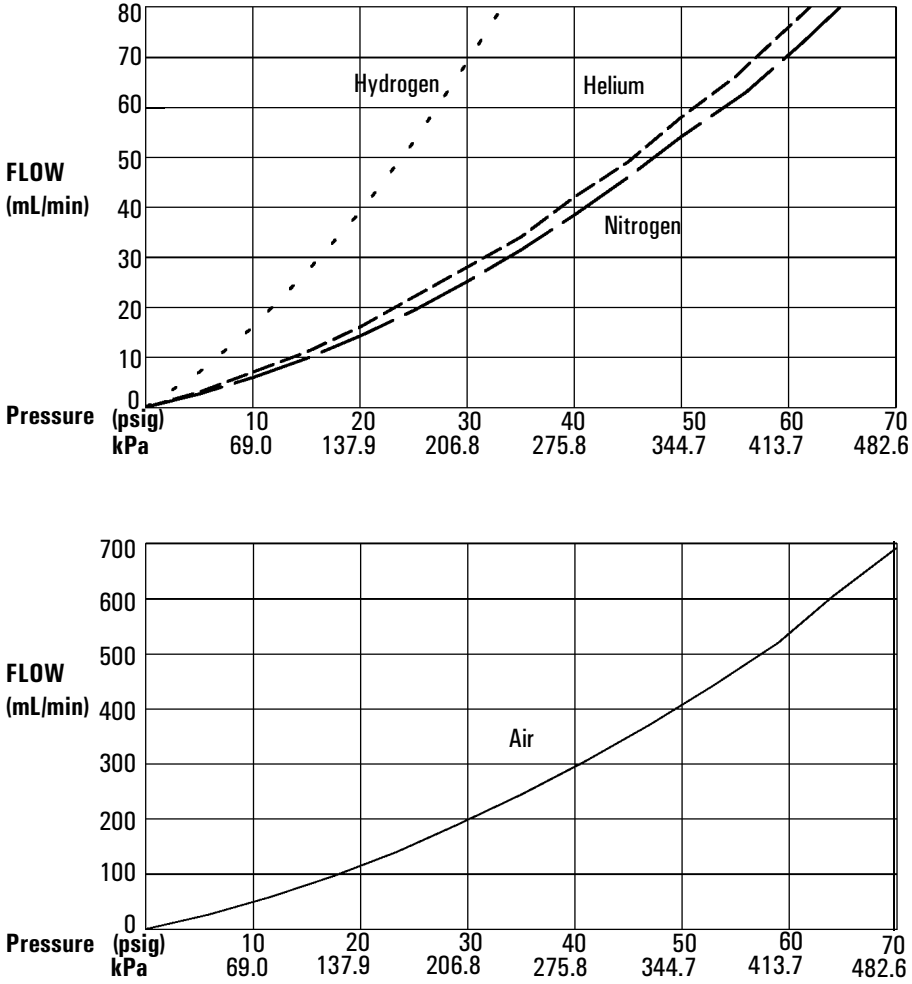
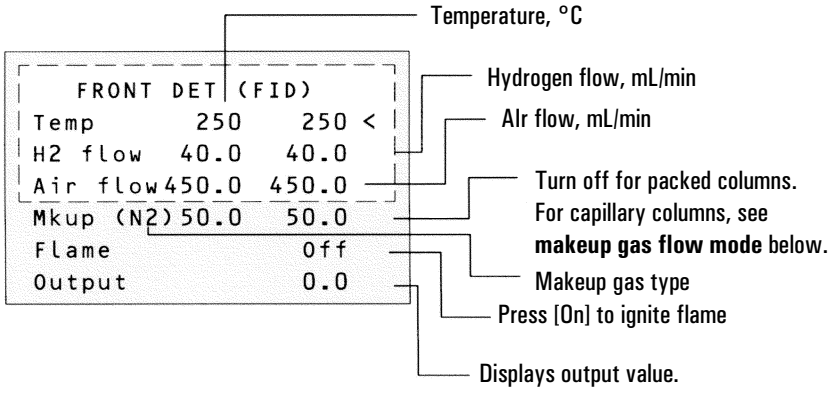


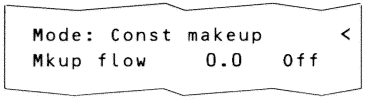
Figure 3. Typical pressure/flow relationships for FID gases
(at 25°C and 1 atmosphere of pressure)

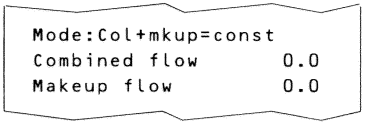
Operating with EPC

Press [Front Det] or [Back Det].

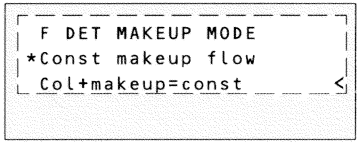


Makeup gas flow mode:
If column dimensions are specified and you have an EPC inlet, the control table will also include one of these:

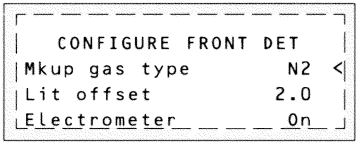




To **change makeup mode,** scroll to Mode: and press [Mode/Type]. Make a selection and enter the appropriate flow values.

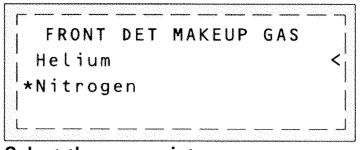


To view **makeup gas** or change **Lit offset,** press [Config][Front Det] or [Config][Back Det]:



It is not necessary to turn the electrometer on or off unless you are performing a maintenance procedure.

To change **makeup gas type,** press [Mode/Type]:



Select the appropriate gas.

Figure 4. FID control table—Electronic Pneumatic Control

Procedure: Using the FID with EPC

Verify that all detector gases are connected, a column is installed, the correct jet is installed, and the system is free of leaks. Check the oven temperature, inlet temperature, and column flow. Use Figure 4 as a guide when operating the FID.

WARNING Verify that a column is installed or the FID column fitting is plugged before turning on the air or hydrogen. An explosion may occur if air and hydrogen are allowed to leak into the oven.

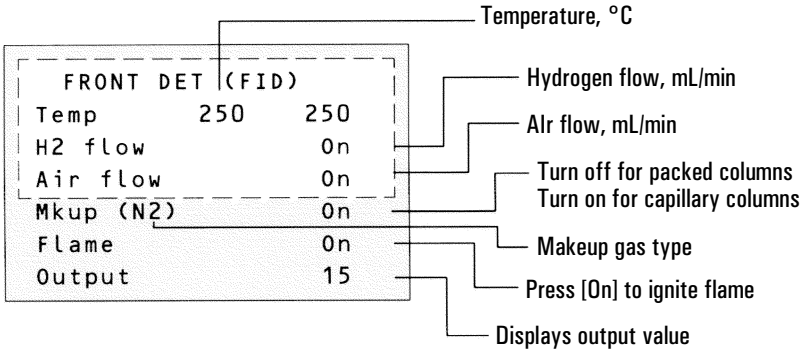
1. Press [Front Det] or [Back Det] to open the FID control table.
2. Set the detector temperature. The temperature must be greater than 150°C for the flame to light.
3. Change the hydrogen flow rate, if desired, and press [Off].
4. Change the air flow rate, if desired, and press [Off].
5. If you are using ***packed columns***, turn off the makeup gas and proceed to Step 7.
6. If you are using ***capillary columns***:
   a. Verify that makeup gas type is the same as that plumbed to your instrument (next to `Mkup` line of control table). Change the gas type, if necessary.
   b. If your capillary column is *defined* and connected to an EPC inlet, choose a new flow mode, if desired, and set the makeup gas flow or combined flow.
   c. If your capillary column is *not defined* or connected to a nonEPC inlet, enter a makeup gas flow. Only constant flow is available in this case.
7. Scroll to `Flame` and press [On]. This turns on the air and hydrogen and initiates the ignition sequence. The signal typically increases to 5 to 20 pA after ignition. Verify that the flame is lit by holding a cold, shiny surface, such as a mirror or chrome-plated wrench, over the collector exit. Steady condensation indicates that the flame is lit.

Short-cut procedure:
(assumes correct setpoints are stored)

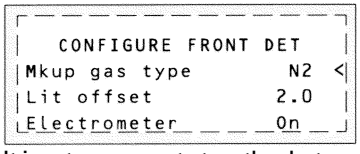
1. Open detector control table.
2. Turn temperature On.
3. Turn makeup gas On, if needed.
4. Press [Det Control].
5. Press [On].

Operating without EPC

Press [Front Det] or [Back Det].

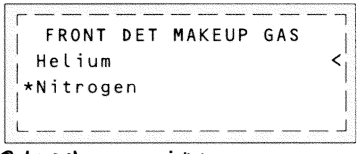


To view **makeup gas** or change **Lit offset,** press [Config][Front Det] or [Config][Back Det]:



It is not necessary to turn the electrometer on or off unless you are performing a maintenance procedure.

To change **makeup gas** type, press [Mode/Type]:



Select the appropriate gas.

Figure 5. FID control table—NonEPC

Procedure: Using the FID without EPC

Verify that detector gases are connected, a column is installed, the correct jet is installed, and the system is free of leaks. Set the oven temperature, inlet temperature, and the inlet or column flow.

1. Press [Front Det] or [Back Det] to access the FID control table. Use Figure 5 as a guide when editing the table.
2. Set the detector temperature.

WARNING Make sure that a column is installed or the FID column fitting is plugged before turning on the air or hydrogen. An explosion may occur if air and hydrogen are allowed to leak into the oven.
Never measure air and hydrogen together. Measure them separately.

Short-cut procedure: *(assumes correct flows are set and temperature setpoint is stored)*

1. Open detector control table.
2. Turn temperature On.
3. Turn makeup gas On, if needed.
4. Press [Det Control].
5. Press [On].

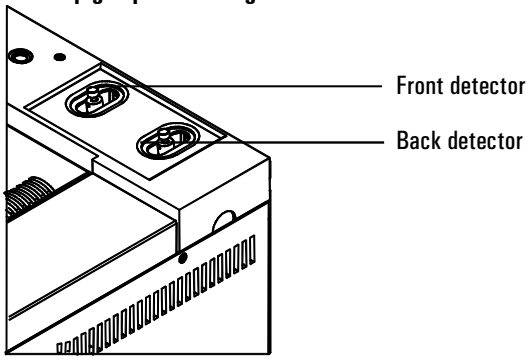
3. Adjust the hydrogen flow.
   a. Make certain the air and makeup gas are turned off.
   b. Turn the hydrogen flow on.
   c. Set the supply pressure, wait for stabilization, and measure flow.
   d. Repeat step 3c until the hydrogen flow is correct. If you have flow through your column, be sure to subtract it from the total flow.
   e. Turn off the hydrogen from the control table while you measure air flow.
4. Adjust the air flow.
   a. Make sure the hydrogen and makeup gas are turned off.
   b. Turn the air flow on.
   c. Set the supply pressure, wait for stabilization, and measure flow.
   d. Repeat step 4c until the air flow is correct. If you have flow through your column, subtract it from the total flow.
   e. Turn the air flow off.

If you are using ***packed columns***, turn the makeup gas off and proceed to step 6.

If you are using ***capillary columns***, verify that the makeup gas type is the same as that plumbed to your instrument (next to `Mkup` line of control table). Change the gas type, if necessary.

5. Set the makeup gas flow.
   a. Make sure the air and hydrogen are turned off.
   b. Set the makeup gas supply pressure.
   c. Locate the knob that controls the pressure regulator on the flow manifold. Turn the knob *clockwise* to increase flow and *counterclockwise* to decrease flow.

Makeup gas pressure regulator



   d. Measure the flow using a flow meter.
   e. Adjust the regulator until the makeup flow is correct. If you have flow going through your column, subtract it from the total flow.
6. Scroll to `Flame` and press [On]. This turns on the air and hydrogen and initiates the ignition sequence. The signal typically increases to 5 to 20 pA after ignition. Verify that the flame is lit by holding a cold, shiny surface, such as a mirror or chrome-plated wrench, over the collector exit. Steady condensation indicates that the flame is lit.

Part 3. Checkout Conditions and Chromatogram

This section contains a typical examples of a test sample chromatogram. It may be used as a general guide to instrument performance.

Note that injection volumes listed with operating conditions do not necessarily indicate total absolute volume injected. Volume given is simply the graduation (plunger position) read from a standard 10 µL syringe. For a heated inlet, actual sample volume injected will also include an additional 0.4-0.7 µL, the volume of sample volatilized from inside the syringe needle. For the dedicated, on-column inlet (unheated), the syringe plunger position more accurately reflects the true injected volume.

Also note that the following procedure and results are intended only to provide evidence of a properly functioning inlet and/or detector system; they are not necessarily suitable to test a given system against its specification limits.

FID checkout conditions

Column and sample

Type	5 30m x 0.32mm x 0.25μm PN 19091J-413
Sample	FID Checkout 18710-60170
Injection volume	1 μL

Inlet

Temperature	250°C Purged/Packed or Split/Splitless
	Oven Track Cool On-Column
	40°C PTV (see below)
Inlet pressure	25 psi (Constant pressure for EPC inlets, helium)

Split/Splitless

Mode	Splitless
Purge flow	60 mL/min
Purge time	0.75 min

Inlet, continued

PTV

Mode	Splitless
Inlet temperature	40°C
Initial time	0.1 min
Rate 1	720°C/min
Final temp 1	350°C
Final time 1	2 min
Rate 2	100°C/min
Final temp 2	250°C
Final time 2	0 min
Inlet pressure	25 psi (Constant pressure for EPC inlets)
Purge time	0.75 min
Purge flow	60 mL/min

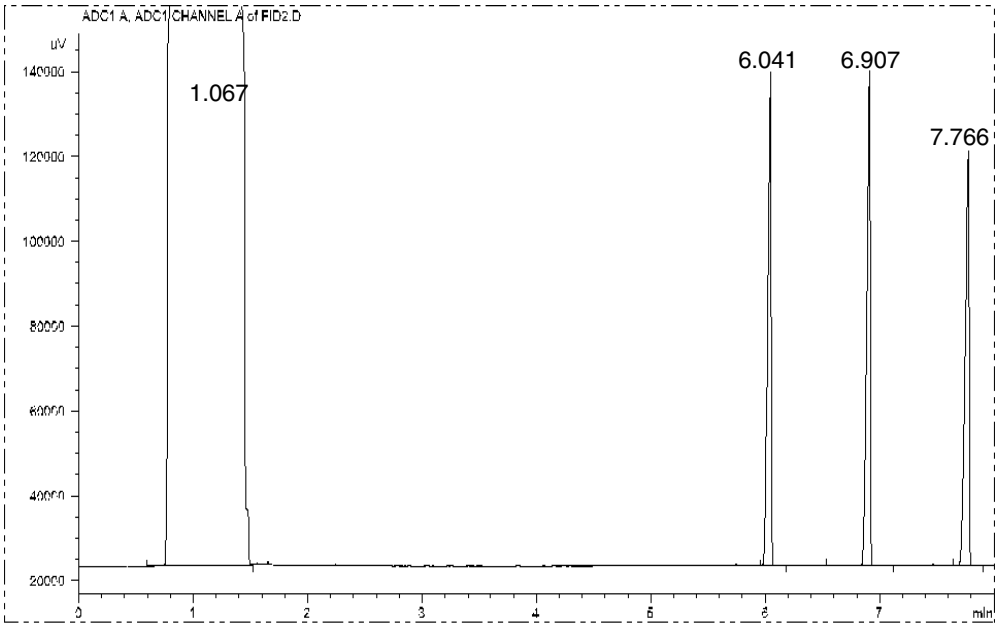
Detector

Temperature	300°C
H_2 flow	30 mL/min
Air flow	400 mL/min
Makeup flow (N_2)	25 mL/min
Offset	Should be < 20 pA

Oven

Initial temp	40°C
Initial time	0 min
Rate 1	25°C/min
Final temp	90°C
Final time	0 min
Rate 2	15°C/min
Final temp	170°C
Final time	2 min

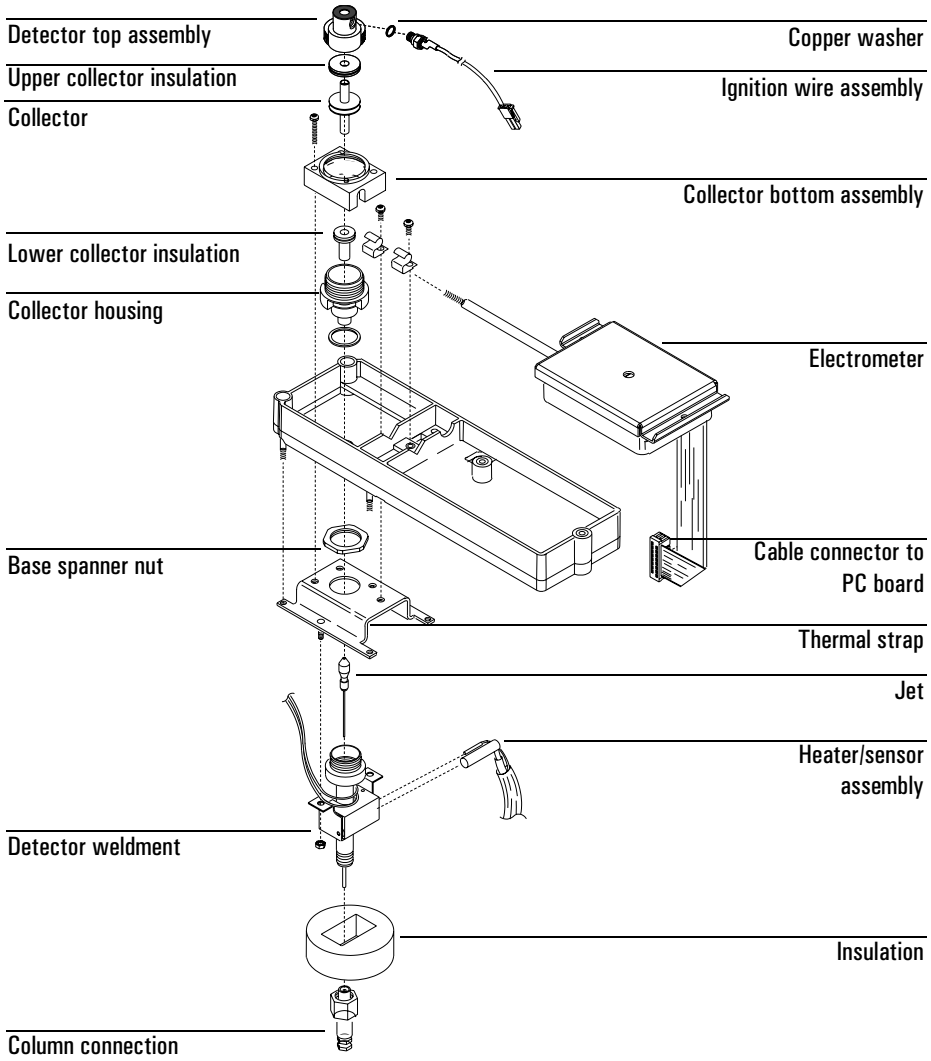
Typical FID checkout chromatogram



Your retention times will differ, but the peaks should be symmetric as in this example.

Part 4. Maintaining a Flame Ionization Detector

WARNING Flame ionization detectors use hydrogen gas as fuel. If hydrogen flow is on and no column is connected to the detector inlet fitting, hydrogen gas can flow into the oven and create an explosion hazard. Detector fittings must have either a column or a cap connected at all times.



Correcting FID hardware problems

The flame goes out or will not light

- Check the column flow rate. It may be too high. Decrease the flow rate or pressure. Switch to a more restrictive column (longer or with a smaller id). If you must use a large id column, turn off the carrier flow long enough to allow the FID to light. Check for partially or completely plugged jet.
- Check that the right type of jet is installed for the column you are using. Jet types are listed on page 14.
- Injecting large volumes of aromatic solvent can cause the flame to go out. Switch to a nonaromatic solvent.
- The lit offset value may be too low or too high. Adjust the value.

WARNING Flame ionization detectors use hydrogen gas as fuel. If hydrogen flow is on and no column is connected to the detector inlet fitting, hydrogen gas can flow into the oven and create an explosion hazard. Detector fittings must have either a column or a cap connected at all times.

Replacing or cleaning the jet

Jets require periodic cleaning or replacement. Even with normal use, deposits develop in the jet (usually white silica from column bleed or black, carbonaceous soot). These deposits reduce sensitivity and cause chromatographic noise and spikes. Although you can clean the jet, it is usually more practical to replace dirty jets with new ones. If you do clean the jet, be very careful not to damage it.

You may also need to change the jet when you change columns or analyses. For example, packed columns use different jets than capillary columns. You must install the proper jet *before* changing the column.

To change a jet, you must first remove the FID collector assembly. The procedure is divided into three parts: removing and inspecting the jet, cleaning the jet (optional), and installing the jet.

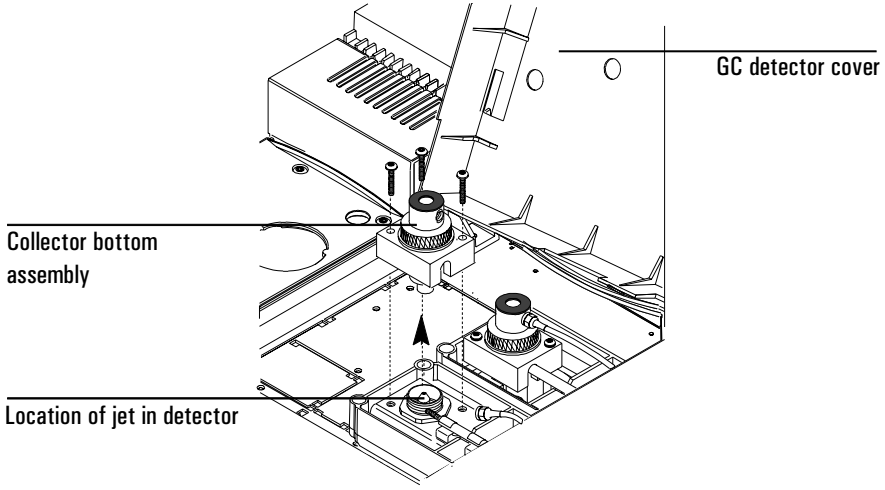
Procedure: Removing and inspecting the jet

Materials needed:

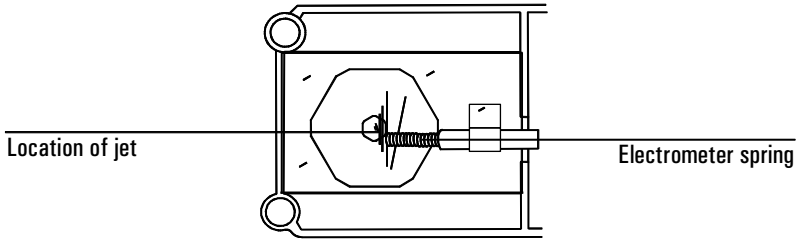
- Gloves to protect hands if detector is hot
- T-20 Torx screwdriver
- 1/4-inch nut driver
- Forceps (or tweezers)

1. Complete the following preliminary steps:
   - Cool the detector to room temperature.
   - When the detector is cool, turn it off and turn off the gases at the GC keyboard.
   - Turn off the electrometer; press [Config] [Front Det] or [Config] [Back Det] to access the control table.
   - Cool the inlet and then turn off the inlet gas.
   - Cool the oven, remove the column, and plug the column connection. See "Installing Columns" in the *General Information* volume for instructions.
   - Open the GC detector cover to access the FID.

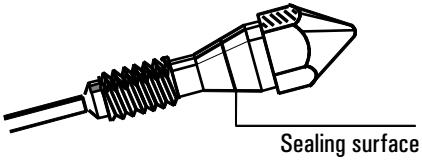
2. Put the gloves on if the detector is hot. Remove the three screws holding the collector bottom assembly in place. Lift off the assembly. The insulator can remain in the collector bottom.



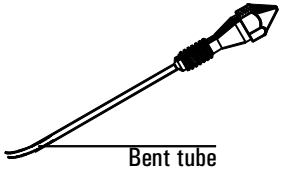
3. Using the nut driver, loosen the jet, and pull it straight out. You may need to use the forceps to grasp the jet.



4. Inspect the jet sealing surface for scratches. You should see a ring around the sealing surface; any other scratches, however, are unacceptable.



5. Inspect the jet tube to make sure it is not bent or crimped. Inspect the jet for contamination or pieces of broken column by holding it up to a light and looking through it. If no contamination is present, the tube will be clear.



Procedure: Cleaning the jet

It is often more convenient to replace dirty jets with new ones than to clean them, especially jets that have been badly contaminated.

If you choose to clean a jet, be careful when using a cleaning wire. Be sure not to scratch the jet internally, because doing so will ruin it. You may want to skip cleaning the jet with a wire and use the aqueous bath only.

Materials needed:

- Small ultrasonic cleaning bath
- Aqueous detergent
- GC-grade methanol in a Teflon wash bottle
- Flame detector cleaning kit (part no. 9301-0985)
- Dry, filtered, compressed air or nitrogen
- Forceps or tweezers

1. Run a cleaning wire through the top of the jet. Run it back and forth a few times until it moves smoothly. Be careful not to scratch the jet.
2. Aqueous cleaning procedure:
    a. Fill the ultrasonic cleaning bath with aqueous detergent and place the jet in the bath. Sonicate for 5 minutes.
    b. Use a jet reamer to clean the inside of the jet.
    c. Sonicate again for 5 minutes.
    From this point on, handle the parts only with forceps (or tweezers)!
    d. Remove the jet from the bath and rinse it thoroughly with hot tap water and then with a small amount of methanol.
    e. Blow the jet dry with a burst of compressed air or nitrogen and then place the jet on a paper towel to air dry.

Procedure: Installing the jet

Caution Do not over-tighten the jet! Over-tightening may permanently deform and damage the jet, the detector base, or both.

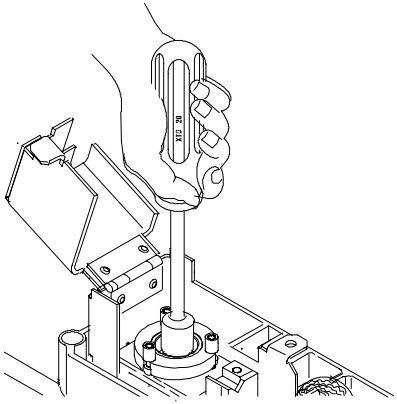
Caution Handle the clean or new jet only with forceps!

Materials needed:

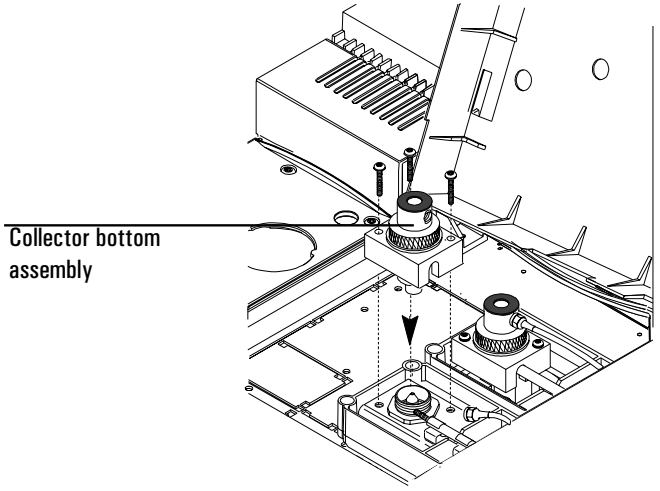
- Gloves to protect hands if detector is hot
- Forceps
- 1/4-inch hex driver
- T-20 Torx screwdriver

See page 14 for tables of jet types.

1. Insert the jet and tighten with the hex driver until it is snug.



2. Replace the collector assembly. Tighten the three screws securing the collector assembly.



3. Reattach the column to the detector. You can now restore normal operating conditions.

Cleaning the collector

The collector requires occasional cleaning to remove deposits (usually white silica from column bleed, or black, carbonaceous soot). Deposits reduce sensitivity and cause chromatographic noise and spikes.

The cleaning procedure presented here suggests you use an ultrasonic bath to clean the collector and other parts of the detector. However, if your collector is not too dirty, it may be sufficient to scrub it with a nylon brush and then use a burst of compressed air or nitrogen to blow stray particles away.

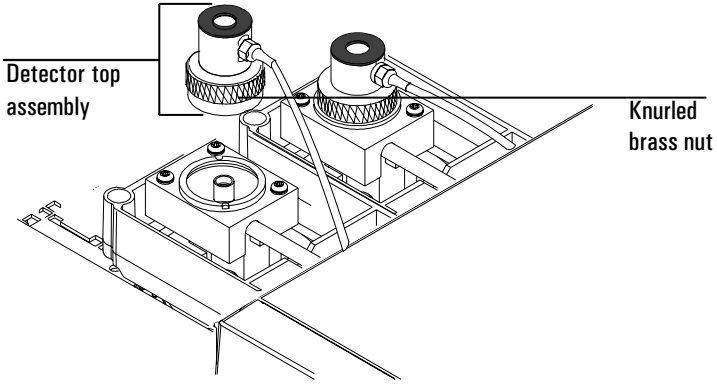
This procedure is divided into three steps: removing the collector, cleaning the collector, and reassembling the detector.

Procedure: Removing the collector

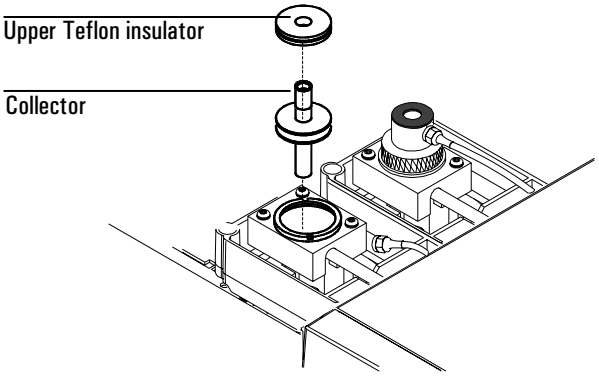
Materials needed:

- T-20 Torx screwdriver
- 1/4-inch nut driver
- Forceps or tweezers
- Gloves if the detector is hot

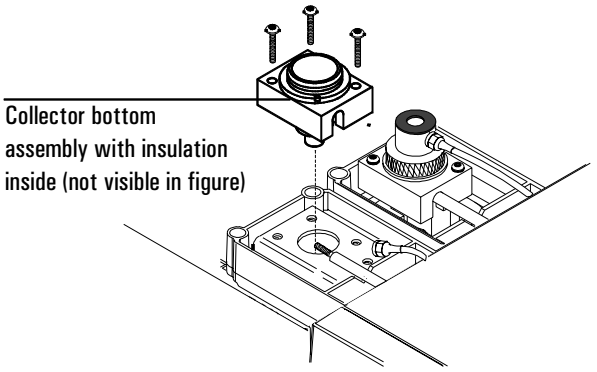
1. Complete the following preliminary steps:
   - Cool the detector to room temperature.
   - When the detector is cool, turn off the temperature zone and the gases at the GC keyboard.
   - Turn off the electrometer; the electrometer control is in the Config Det table. Press [Config] [Front Det] or [Config] [Back Det] to access the control table.
   - Open the GC detector cover to access the FID.
2. Put on the gloves if the detector is hot. Loosen the knurled brass nut. Lift the top assembly straight up. The upper Teflon insulator might stick to the bottom of the assembly. Remove the insulator.



3. Lift out the collector. The upper insulator may be attached to the collector. You may need to use the tweezers to grasp the collector.



4. Remove the three screws that hold the collector bottom assembly in place. Lift off the assembly. Remove the lower insulator from the bottom assembly. You may need to use the forceps to grab it.



Procedure: Cleaning the collector

Materials needed:

- Small ultrasonic cleaning bath
- Aqueous detergent
- GC-grade methanol in a Teflon wash bottle
- Flame detector cleaning kit (part no. 9301-0985)
- Dry, filtered, compressed air or nitrogen
- Forceps or tweezers

Cleaning procedure:

1. Fill the ultrasonic cleaning bath with aqueous detergent, and place the two insulators and the collector in the bath. Sonicate for 5 minutes.
2. Use the nylon brushes to clean each piece.
3. Sonicate again for 5 minutes.
   From this point on, handle the parts only with forceps or tweezers!
4. Remove the pieces from the bath and rinse them thoroughly with hot tap water and then with a small amount of methanol.
5. Place the pieces on a paper towel to air dry.

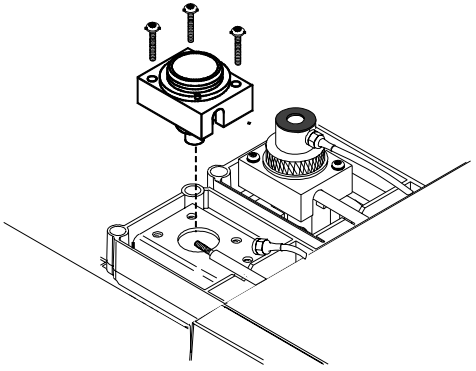
Procedure: Reassembling the detector

Caution Handle the clean collector and insulators only with forceps (or tweezers)!

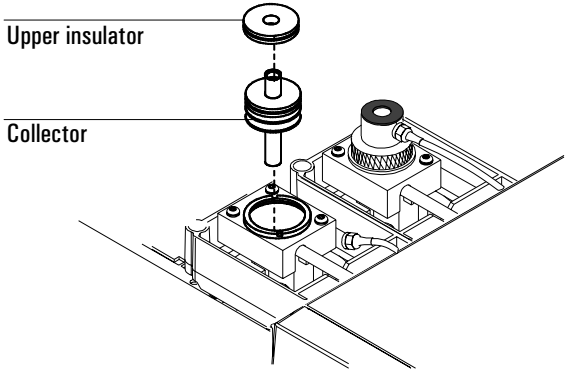
Materials needed:

- Forceps or tweezers
- T-20 Torx screwdriver

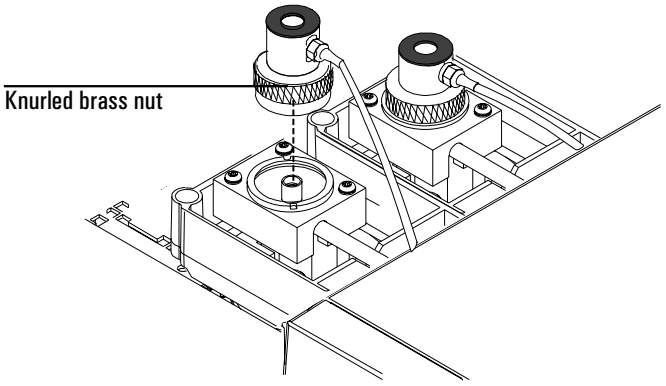
1. Insert the lower insulator into the lower collector assembly. Install the lower collector assembly and tighten the three screws.



2. Replace the collector and install the upper Teflon insulator.



3. Install the upper collector assembly and tighten the knurled nut finger-tight.



4. Close the GC detector cover. You can now restore normal operating conditions.

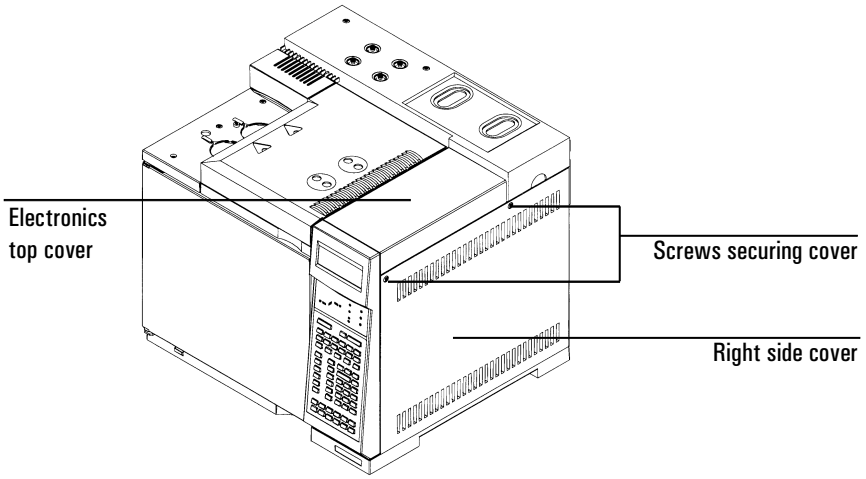
Procedure: Replacing the FID ignition wire

Materials needed:

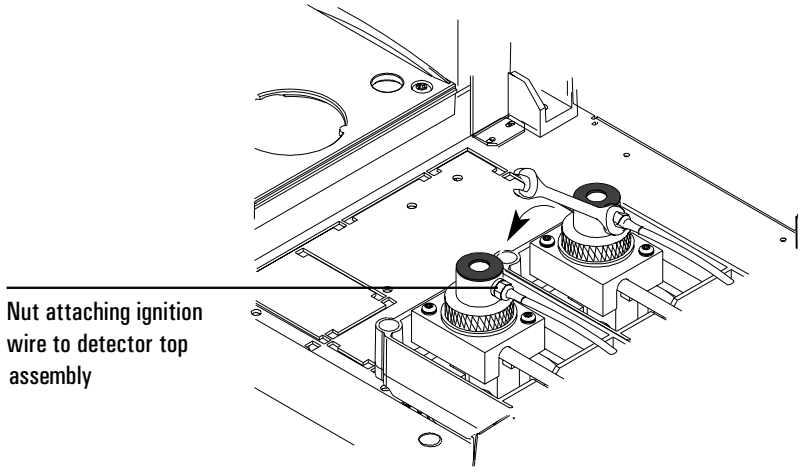
- 5/16-inch wrench
- T-20 Torx screwdriver
- ESD wrist strap
- New ignition wire assembly (part no. G1531-60680)

1. Complete the following preliminary steps:
    - Allow the detector to cool to room temperature. When the detector is cool, turn off the GC.
    - Lift the GC detector cover to access the FID.
    - Remove the electronics top cover.

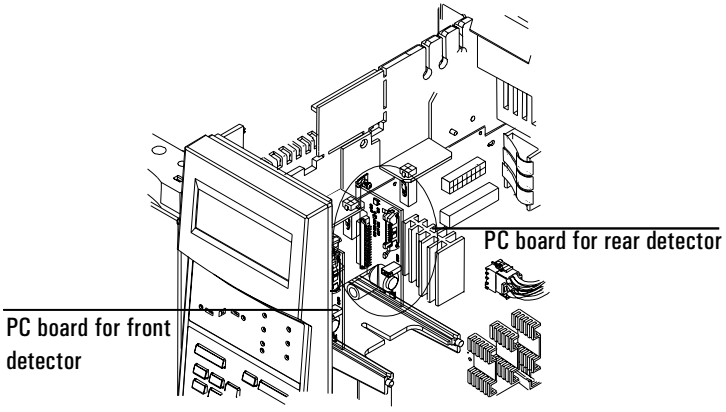
2. Remove the two screws securing the right side cover and remove the cover.



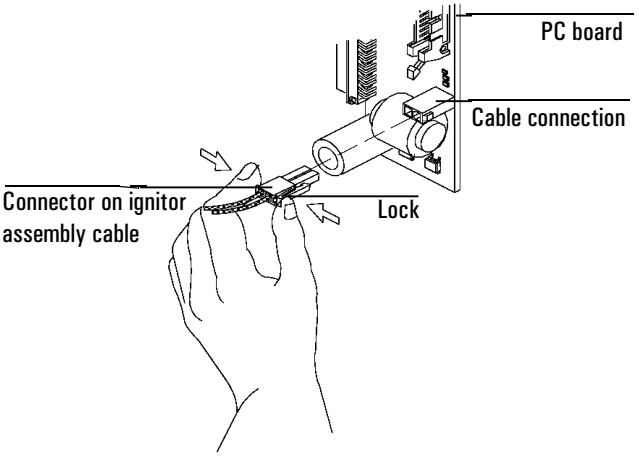
3. Using the wrench, loosen the ignition wire from the detector top assembly. Disconnect the wire completely. Do not lose the small copper washer between the top assembly and the ignition wire connection.



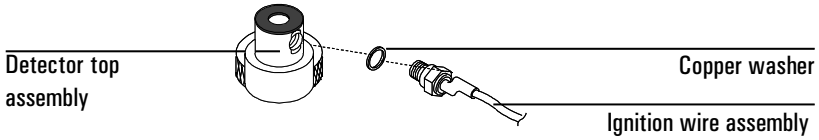
4. The other end of the ignition cable is connected to the detector PC board. Use the figure below to locate the PCB. Make sure to put on the ESD wrist strap at this time and connect it to a proper ground.



5. To disconnect the cable connection, squeeze the lock and gently pull the connector free. Attach the new ignitor cable by squeezing the lock and sliding the connector into the slot.



6. Place the copper washer on the other end of the ignition cable. Attach the other end of the ignition cable to the detector top assembly, and finger-tighten the screw to snugness. Then use the screwdriver to tighten the screw firmly.

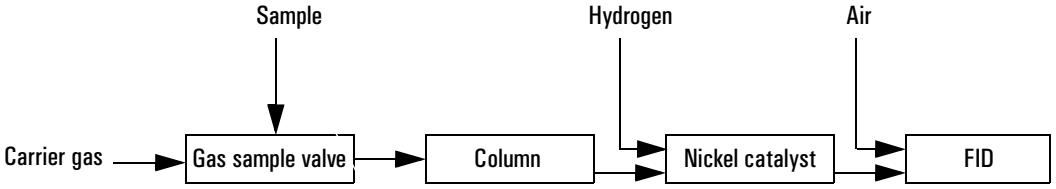


7. Replace the right side cover and the two screws. Replace the electronics top cover.

8. Turn on the GC and restore normal operating conditions.

Part 5. The Nickel Catalyst Tube

The Nickel Catalyst Tube accessory, G2747A, is used for trace analysis of CO and CO_2 with a flame ionization detector. The gas sample is separated on the column and passed over the hot catalyst in the presence of hydrogen, which converts the CO and CO_2 peaks to CH_4.



Gas flows

For a standard FID installation:

Gas	Flow rate, mL/min
Carrier (helium)	30
FID hydrogen	30 (see Caution)
FID air	400

For a TCD/FID in-series installation:

Gas	Flow rate, mL/min
Carrier (helium)	30
TCD switching flow	25
FID hydrogen	45 (see Caution)
FID air	500

Caution Hydrogen flow is pressure-controlled, where an FID provides a known resistance. The nickel catalyst tube increases flow resistance, so that the calibration is no longer valid. You must measure hydrogen flow with a bubble or similar meter. See your GC Operating Manual for details.

Caution The nickel catalyst can be damaged by exposure to air.

Temperature

The nickel catalyst tube is usually mounted in the back inlet position and controlled by the back inlet temperature setpoint. For most analyses, set these temperatures:

- Nickel catalyst tube 375°C
- FID 400°C

Repacking the catalyst

The nickel catalyst can be damaged by exposure to air or by impurities in the samples or gases. If performance is significantly degraded, repack the catalyst tube.

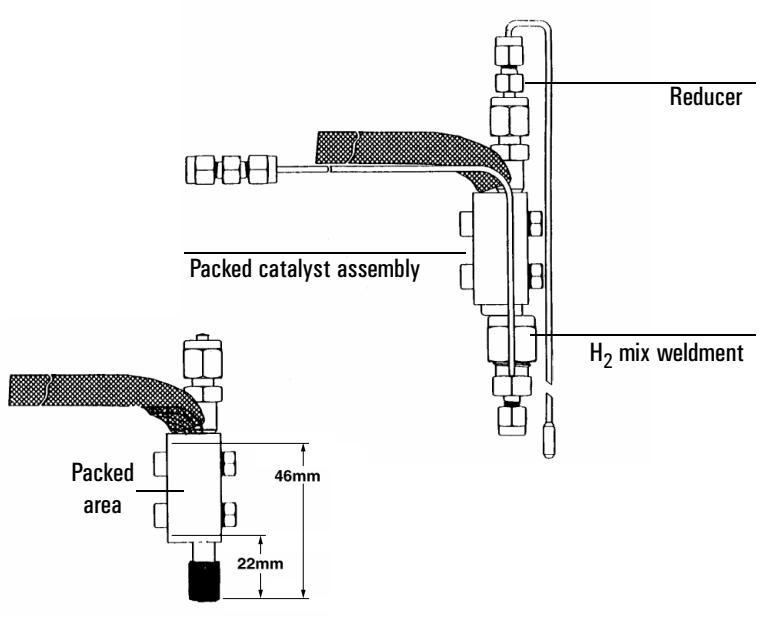
WARNING Hydrogen (H_2) is flammable and is an explosion hazard when mixed with air in an enclosed space (for example, the oven). In any application using H_2, turn off the supply at its source before working on the instrument.

WARNING Both nickel oxide and some forms of silicon oxide are considered carcinogens for humans. Perform all work in a fume hood and wear cotton gloves at all times. Remove any spills with an HEPA-type vacuum cleaner, avoiding any action that raises dust. Alert your company's Safety group if a spill occurs.

WARNING Due to the possibility of dermatitis, wash the arms and hands with soap and water after use. Long sleeves are recommended during any use and spill cleanup. If long sleeves are not worn, long gloves are an acceptable substitute.

Caution Be sure to read the Material Safety Data Sheet (MSDS) provided with the catalyst before performing this procedure.

1. Turn off the back inlet thermal zone. Turn off all other heaters. When the catalyst tube has cooled to room temperature, turn off the power to the GC and disconnect the power cord. Bleed down the residual hydrogen and carrier gas pressures.
2. Remove the three screws holding the cover plate on top of the catalyst tube. Remove the plate and the insulation around the NCT.
3. From inside the oven, loosen the two screws holding the insulation cup. Remove the cup and insulation.
4. Use two wrenches to disconnect the H_2 mix weldment from the bottom of the catalyst assembly. Be careful NOT to place stress on the 1/16-inch tube. Stress can damage the weldment.



5. Use two wrenches to remove the reducer on the top of the catalyst assembly.
6. Gently lift the catalyst assembly out of the injection area. Both ends of the catalyst tube are now accessible.
7. Use a hooked instrument to remove the glass wool plug from the bottom of the tube. Make sure you get all of it.

8. Empty the old catalyst from the tube (you may have to break it out with a pointed tool). Make sure you get it all out.
9. Use a thin rod to push out the top glass wool plug from the tube.
10. Clean the inside of the tube thoroughly with methanol. Do not use any sharp metal tools on the inside of the tube. A cotton swab carefully used will ensure cleanliness. Dry the tube.
11. The previous figure shows the dimensions for repacking the tube correctly. If any catalyst is outside the heated zone, severe tailing of CO will result.

    Prepare a simple depth gauge using a wooden cotton swab or any other handy rod or tubing. Use tape or paint to mark the stick at 46 mm from the blunt end and at 22 mm from the blunt end.
12. Roll up a piece of glass wool about the size of a large pea. Push this into the tube from the 1/4-inch end and seat it firmly. Measure the depth of this glass wool with the depth gauge—it should be 46 mm from the end of the tube. If necessary, add more glass wool. A slight compression of the glass wool during the measurement works best.
13. Turn the catalyst assembly upside down and add catalyst slowly. Tap gently to help seat it. When the catalyst is 22 mm from the end, stop adding catalyst. Do NOT crush the catalyst when packing or measuring the depth.
14. Add a single glass wool plug to fill the remaining part of the tube to within 5 mm of the end. This plug should be gently compressed during installation.

Caution Before installing the catalyst assembly into the oven, carefully wipe it to remove any catalyst dust.

15. Reassembly is the reverse of steps 1 through 6. Make sure that the insulation is carefully repacked around the tube before you reinstall the injector cover plate and the insulation cup.
16. Leak test the new installation.

WARNING Hydrogen (H_2) is flammable and is an explosion hazard when mixed with air and confined in an enclosed space (for example, the oven).

17. Start the carrier and hydrogen flows. Allow them to flow for 15 minutes.
18. Heat the nickel catalyst to 375°C and hold for 30 minutes. The accessory is ready for use.

3

The Thermal Conductivity Detector

Chapter 3
The Thermal Conductivity Detector

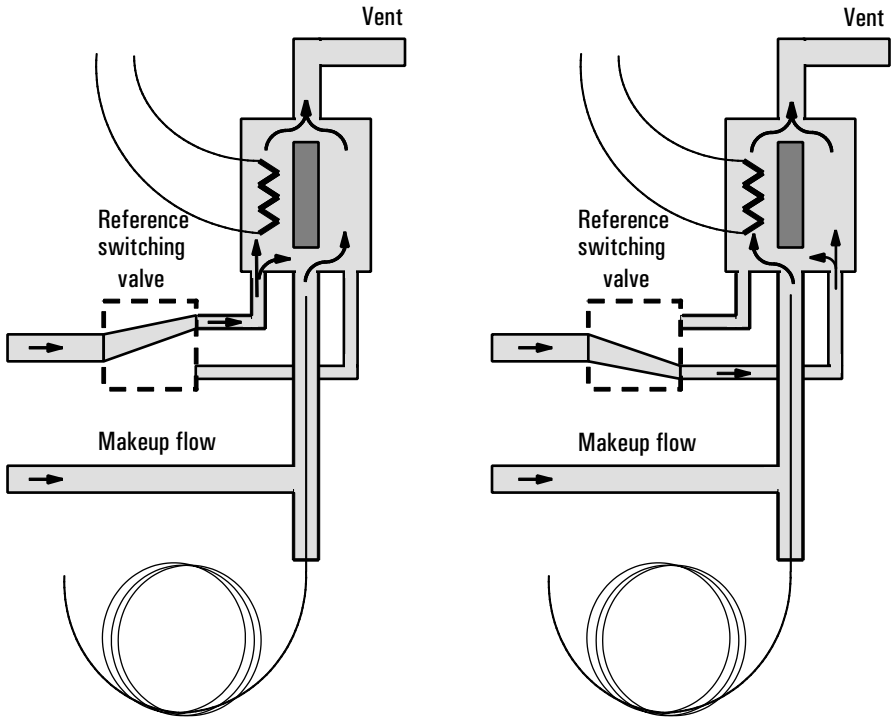
Part 1. General Information

The TCD compares the thermal conductivities of two gas flows—pure carrier gas (also called the reference gas) and carrier gas plus sample components (also called column effluent).

This detector contains a filament that is heated electrically so that it is hotter than the detector body. The filament temperature is kept constant while alternate streams of reference gas and column effluent pass over it. When sample is added, the power required to keep the filament temperature constant changes. The two gas streams are switched over the filament five times per second and the power differences are measured and recorded.

When helium (or hydrogen) is used as carrier gas, the sample causes the thermal conductivity to fall. If nitrogen is used, the thermal conductivity usually goes up because most things are more conductive than nitrogen.

Because the TCD does not destroy the sample during the detection process, this detector can be hooked up in series to a flame ionization detector or other detector.



Column effluent diverted to bypass channel. Filament surrounded by reference gas.

Column effluent diverted to filament channel. If sample is present, thermal conductivity rises or falls, depending on gas type.

Figure 6. TCD — Conceptual diagram

TCD pneumatics

Figure 7 and Figure 8 show the pneumatics design of the TCD, with and without EPC.

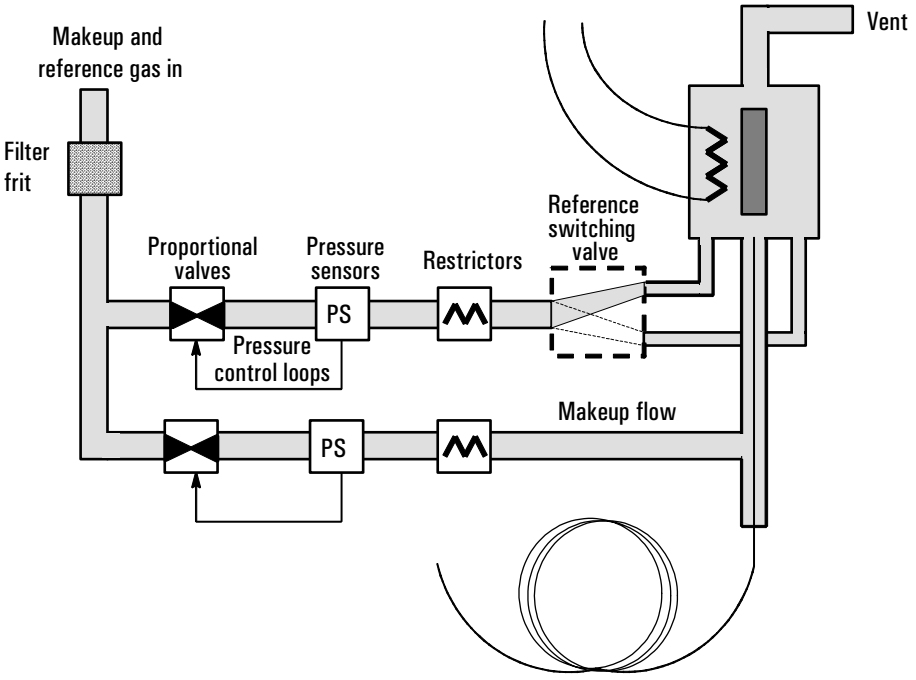


Figure 7. TCD pneumatics—EPC

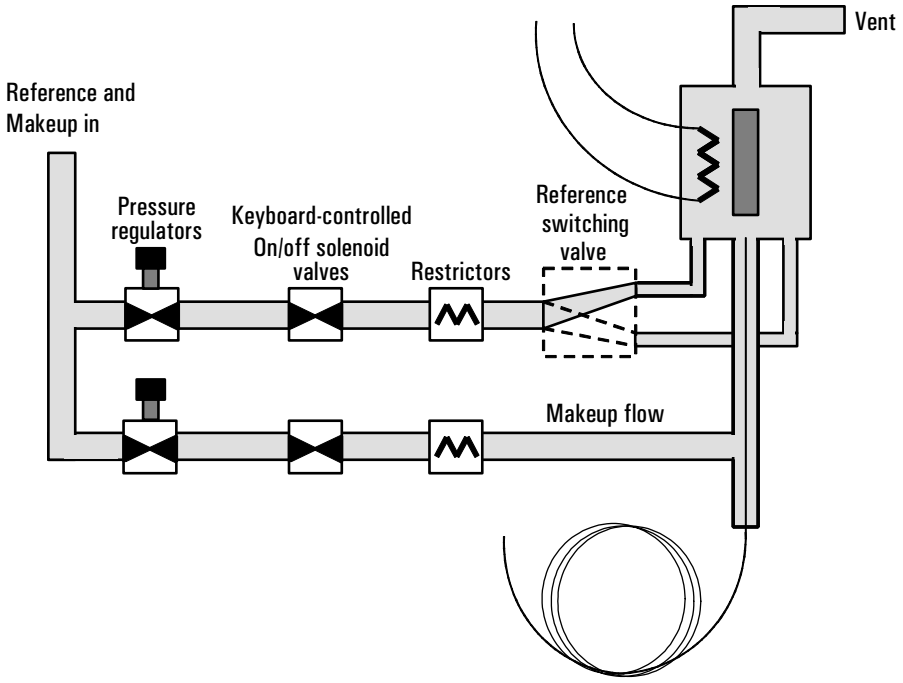


Figure 8. TCD pneumatics—NonEPC

Conditions that prevent the detector from operating

- Temperature set below 150°C
- Broken or shorted filament
- Reference gas flow set to 0

Filament passivation

The tungsten-rhenium TCD filament has been chemically passivated to protect against oxygen damage. However, chemically active compounds such as acids and halogenated compounds may attack the filament. The immediate symptom is a permanent change in detector sensitivity due to a change in filament resistance.

If possible, such compounds should be avoided. If this is not possible, the filament may have to be replaced frequently.

Carrier, reference, and makeup gas

Reference and makeup gas must be the same as the carrier gas, and the gas type must be specified in both the inlet and detector control tables.

When using packed columns, we recommend a small makeup gas flow (2 to 3 mL/min) to get the best peak shapes.

Use Figure 9 to select a value for reference gas flow for either capillary or packed columns. Any ratio within ±0.25 of that in the figure is suitable.

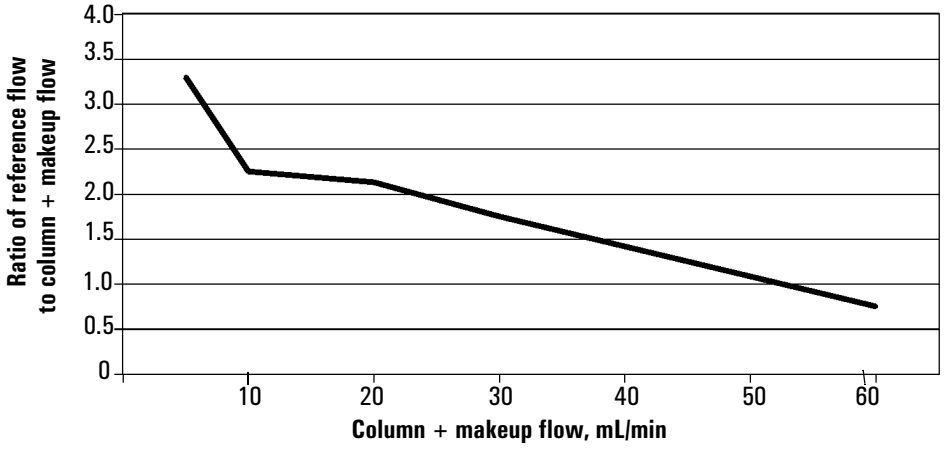


Figure 9. Selecting the reference gas flow

Negative polarity

Sample components with higher thermal conductivities than the carrier gas produce negative peaks. For example, helium or hydrogen form a negative peak with nitrogen or argon-methane as the carrier gas.

`Neg polarity ON` inverts the peak so the integrator or ChemStation can measure it. `Neg polarity` can be a run table entry; see chapter 9, "Instrument Automation", in the *General Information* volume.

Analyzing for hydrogen

Hydrogen is the only element with thermal conductivity greater than helium, and mixtures of small amounts of hydrogen (<20%) in helium at moderate temperatures exhibit thermal conductivities less than either component alone. If you are analyzing for hydrogen with helium carrier gas, a hydrogen peak may appear as positive, negative, or as a split peak.

There are two solutions to this problem:

- Use nitrogen or argon-methane as carrier gas. This eliminates problems inherent with using helium as carrier, but causes reduced sensitivity to components other than hydrogen.
- Operate the detector at higher temperatures—from 200°C to 300°C.

You can find the correct detector operating temperature by analyzing a known range of hydrogen concentrations, increasing the operating temperature until the hydrogen peak exhibits normal shape and is always in the same direction (negative relative to normal response to air or propane) regardless of concentration. This temperature also ensures high sensitivity and linear dynamic range.

Because hydrogen peaks are negative, you must turn negative polarity on at appropriate times so the peak appears positive.

Part 2. Operating the TCD

Use the information in Table 4 when selecting temperatures and flows for the TCD. Use Figure 10 to locate minimum source pressures. If you have an EPC detector, you must add 10 psi (69 kPa) to the source pressure on the chart.

Table 4. Recommended Flow Rates and Temperatures

Gas type	Flow range
Carrier gas *(hydrogen, helium, nitrogen)*	Packed, 10 to 60 mL/min Capillary, 1 to 5 mL/min
Reference *(same gas type as carrier)*	15 to 60 mL/min See Figure 10 to select a value.
Capillary makeup *(same gas type as carrier)*	5 to 15 mL/min—capillary columns 2 to 3 mL/min—packed columns
Detector temperature	
< 150°C, cannot turn on filament	
Detector temperature should be 30°C to 50°C greater than highest oven ramp temperature.	

Gas pressures

NonEPC detector—choose a flow, find a starting source pressure.
EPC detector—choose a flow, find a pressure, set source pressure 10 psi higher.

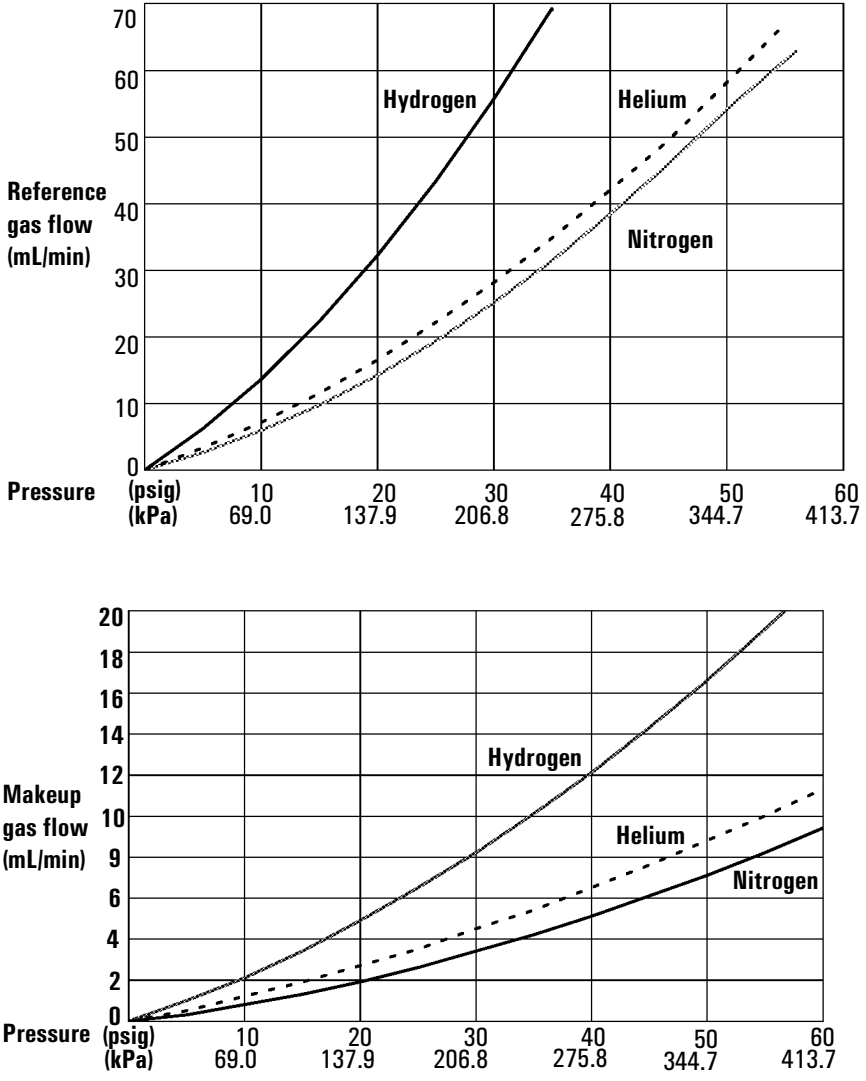
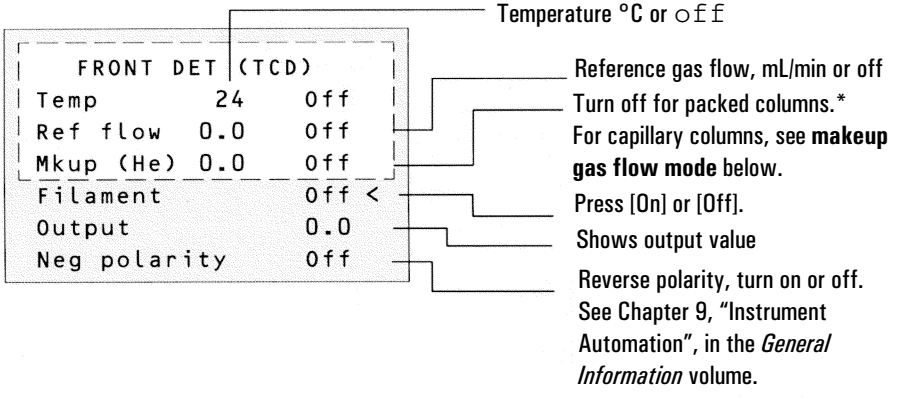


Figure 10. Typical pressure/flow relationships, reference and makeup gases *(at 25°C and 1 atmosphere of pressure)*

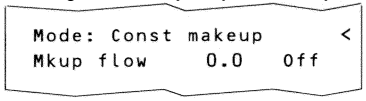
Operating with EPC

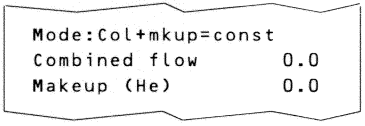
Press [Front Det] or [Back Det].



Makeup gas flow mode:

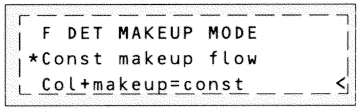
If configured for capillary columns, your control table will also include one of these:



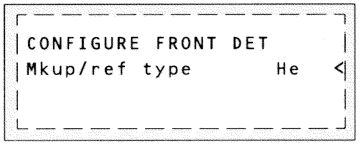


To **change makeup mode,** scroll to Mode: and press [Mode/Type].

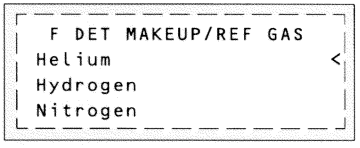
Make a selection and enter the appropriate flow values.



To **view makeup/reference gas,** press [Config][Front Det] or [Config][Back Det]:



To **change makeup/reference gas,** press [Mode/Type]:



Select the appropriate gas.

* A makeup flow of 2 to 3 mL/min improves peak shapes.

Figure 11. TCD control table with EPC

Procedure: Using the TCD with EPC

This procedure assumes that detector support gases are connected, the system is leak-free, and a column is installed. Before operating the detector, set oven temperature, inlet temperature, and inlet/column flow.

1. Press [Front Det] or [Back Det] to open the detector control table.
2. Set the detector temperature. Do not set higher than the maximum temperature allowed for the column because part of the column passes through the heated block and into the cell.
3. Verify that gas type is the same as that plumbed to your instrument (next to `Mkup` line of control table). Change the gas type, if necessary (page 7).

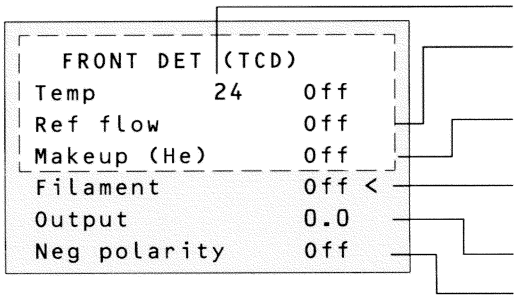
Caution Detector electronics depend on the correct gas type configuration.

4. Set the reference gas flow rate.
5. If you are using ***packed columns***, turn off the makeup gas (or proceed to Step 6 and enter 2 to 3 mL/min, see page 54) and proceed to Step 7.
6. If you are using ***capillary columns:***
   a. If your column is *defined* and connected to an EPC inlet, choose a new flow mode (page 7) if desired, and set the makeup gas flow or combined flow.
   b. If your column is connected to a nonEPC inlet, enter a makeup gas flow. Only constant flow is available in this case.
7. Turn on the filament. Allow about 30 minutes for thermal stabilization. A longer period may be needed for the highest sensitivity.
8. If necessary, turn `Negative polarity` [On] to invert negative-going peaks. When a sample contains components giving both positive- and negative-going peaks, `Neg polarity` can be switched on and off during a run as a timetable event.

Short-cut procedure:
(assumes your setpoints are stored)

1. Open detector control table.
2. Turn temperature `On`.
3. Turn makeup gas `On`, if needed.
4. Press [Det Control]
5. Press [On]

Operating without EPC



Temperature °C

Reference gas flow, turn On or Off, see **makeup/reference gas** below.

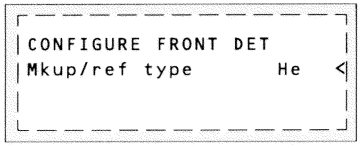
Off for packed columns.
On for capillary columns.

Press [On] or [Off].
Turn on gas flows first.

Shows output value

Reverse polarity, turn on or off.

To **view makeup/reference gas,** press [Config][Front Det] or [Config][Back Det]:



To **change makeup/reference gas,** press [Mode/Type]:

Select the appropriate gas.

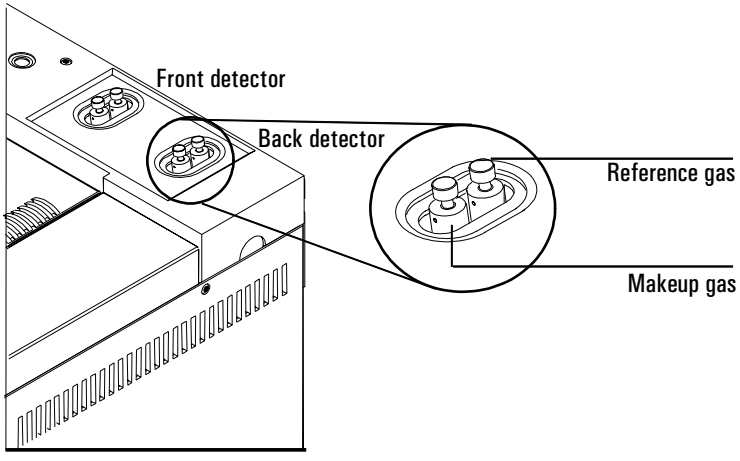
Figure 12. TCD control table without EPC

Procedure: Using the TCD without EPC

This procedure assumes that detector support gases are connected, the system is leak-free, and a column is installed. Before beginning, set the oven temperature and inlet temperature and flow.

Use a bubble flow meter or similar device to measure flows.

1. Press [Front Det] or [Back Det] to access the detector control table.
2. Set the detector temperature. Do not set higher than the maximum temperature allowed for the column, because part of the column passes through the TCD heated block and into the cell.
3. Verify that makeup gas type is the same as that plumbed to your instrument (next to `Mkup` line of control table). Change the gas type, if necessary.
4. Turn the makeup gas flow off from the control table.
5. Turn the `Ref flow` on from the control table.
   a. Set the supply pressure.
   b. Locate the knob that controls the pressure regulator for reference gas on the flow manifold. Turn the knob *clockwise* to increase flow and *counterclockwise* to decrease flow.



c. Measure the flow. Adjust the regulator until the reference flow is correct. If you have flow going through your column, be sure to subtract it from the total flow.

d. Turn off the reference gas at the control table.

6. If you are using ***packed columns*** and are not using makeup gas, turn off the makeup gas from the control table and proceed to step 8..

7. If you are using ***capillary columns*** or ***packed columns with makeup gas***:

   a. Turn makeup gas on from the control table.

   b. Locate the knob that controls the pressure regulator for makeup gas on the flow manifold. Turn the knob clockwise to increase flow and counterclockwise to decrease flow.

   c. Measure the flow and adjust the regulator until the makeup flow is correct. If you have flow going through your column, subtract it from the total flow.

8. Turn the reference flow on from the keyboard.

9. Turn the filament on. Allow about 30 minutes for thermal stabilization before using. A longer period may be required for the highest sensitivity.

10. If necessary, turn `Neg polarity` on to invert negative-going peaks.

    When a sample contains components giving both positive- and negative-going peaks, `Neg polarity` can be switched on and off during a run as a timetable event.

Short-cut procedure:
(assumes correct setpoints are stored and correct flows are set)

1. Open detector control table.
2. Turn temperature `On`.
3. Turn makeup gas `On`, if needed.
4. Press [Det Control].
5. Press [On].

Part 3. Checkout Conditions and Chromatogram

This section contains a typical examples of a test sample chromatogram. It may be used as a general guide to instrument performance.

Note that injection volumes listed with operating conditions do not necessarily indicate total absolute volume injected. Volume given is simply the graduation (plunger position) read from a standard 10 µL syringe. For a heated inlet, actual sample volume injected will also include an additional 0.4-0.7 µL, the volume of sample volatilized from inside the syringe needle. For the dedicated, on-column inlet (unheated), the syringe plunger position more accurately reflects the true injected volume.

Also note that the following procedure and results are intended only to provide evidence of a properly functioning inlet and/or detector system; they are not necessarily suitable to test a given system against its specification limits.

TCD checkout conditions

Column and sample

Type	5 30m × 0.32mm × 0.25 μm PN 19091J-413
Sample	FID Checkout 18710-60170
Injection volume	1 μL

Inlet

Temperature	250°C Purged/Packed or Split/Splitless
	Oven Track Cool On-Column
	40°C PTV (see below)
Inlet pressure	25 psi (Constant pressure for EPC inlets, helium)

Split/Splitless

Mode	Splitless
Purge flow	60 mL/min
Purge time	0.75 min

Inlet, continued

PTV

Mode	Splitless
Inlet temperature	40°C
Initial time	0.1 min
Rate 1	720°C/min
Final temp 1	350°C
Final time 1	2 min
Rate 2	100°C/min
Final temp 2	250°C
Final time 2	0 min
Inlet pressure	25 psi (constant pressure for EPV inlets)
Purge time	0.75 min
Purge flow	60 mL/min

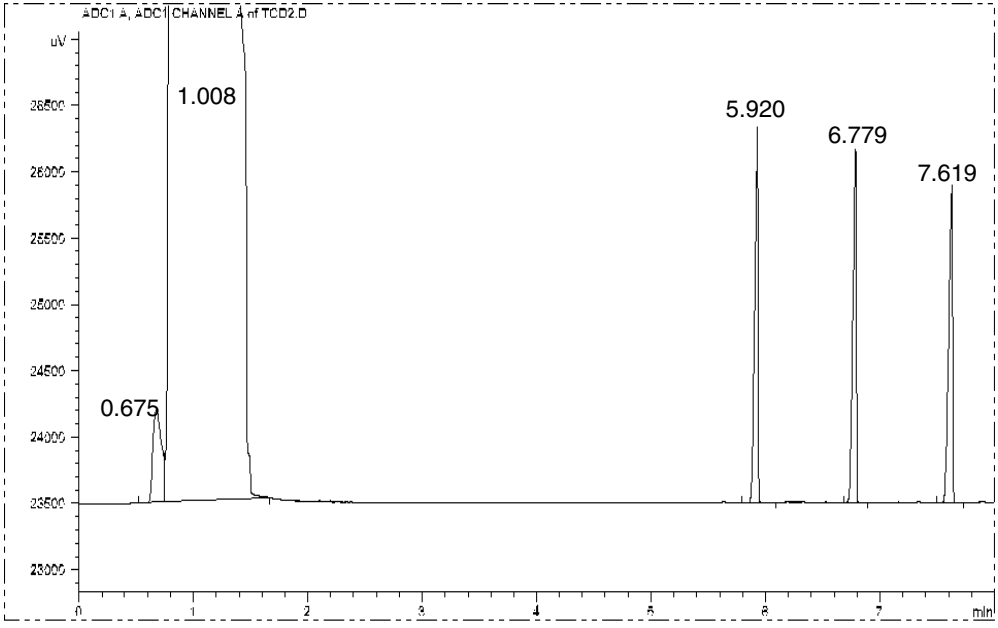
Detector

Temperature	300°C
Reference flow (He)	30 mL/min
Makeup flow (He)	2 mL/min
Offset	Should be < 30 display counts

Oven

Initial temp	40°C
Initial time	0 min
Rate 1	25°C/min
Final temp	90°C
Final time	0 min
Rate 2	15°C/min
Final temp	170°C
Final time	2 min

Typical TCD checkout chromatogram



Your retention times will differ, but peaks should resemble this example.

Part 4. Maintaining a Thermal Conductivity Detector

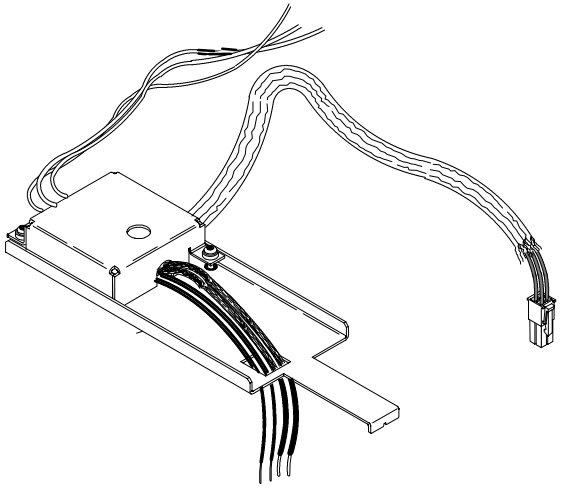


Figure 13. The TCD

Correcting TCD performance problems

If the TCD is displaying problems such as a wandering baseline, increased noise level, or changes in response on a checkout chromatogram, it is probably contaminated with deposits from such things as column bleed or dirty samples.

The TCD is cleaned by a process known as bakeout. Bakeout should be performed only after you have confirmed that the carrier gas and the flow system components are leak and contaminant free.

Caution Baking out the detector with a large air leak present can destroy the filament.

Procedure: Thermal cleaning

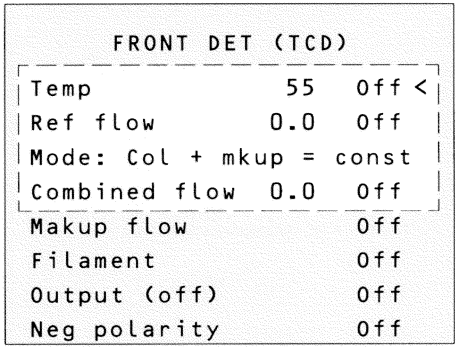
The only common maintenance procedure you will need to perform on the TCD is thermal cleaning.

The TCD can become contaminated with deposits from such things as column bleed or dirty samples. A wandering baseline, increased noise level, or changes in response on a checkout chromatogram all indicate contamination.

Thermal cleaning is also known as **bakeout**. Bakeout should be performed only after you have confirmed that the carrier gas and the flow system components are leak and contaminant free.

Caution You must turn off the TCD and cap the detector column fitting to prevent irreparable damage to the filament caused by oxygen entering the detector.

1. Turn the detector off.
2. Remove the column from the detector and cap the detector column fitting.
3. Set the reference gas flow rate between 20 and 30 mL/min. Set the detector temperature to 400°C.



4. Allow thermal cleaning to continue for several hours. Then cool the system to normal operating temperatures.

4

The Nitrogen-Phosphorus Detector

Chapter 4
The Nitrogen-Phosphorus Detector

Part 1. General Information

The NPD passes sample and carrier through a hydrogen/air plasma. A heated ceramic source, called the bead, is just above the jet. The low hydrogen/air ratio cannot sustain a flame, minimizing hydrocarbon ionization, while the alkali ions on the bead surface facilitate ionization of nitrogen- or phosphorous-organic compounds. The output current is proportional to the number of ions collected. It is sensed by an electrometer, converted to digital form, and sent to an output device.

Software requirements

This discussion assumes that the following firmware/software is installed:

Product	Software/firmware revision
6890A GC	A.03.03 or higher
Agilent GC ChemStation	A.05.02 or higher
Agilent MSD ChemStation	G1701AA or higher

Software/firmware with numbers less than shown in the table will cause reduced bead lifetime. See Agilent service for updates.

NPD pneumatics

Figure 14 and Figure 15 show the flow paths for the NPD with and without EPC.

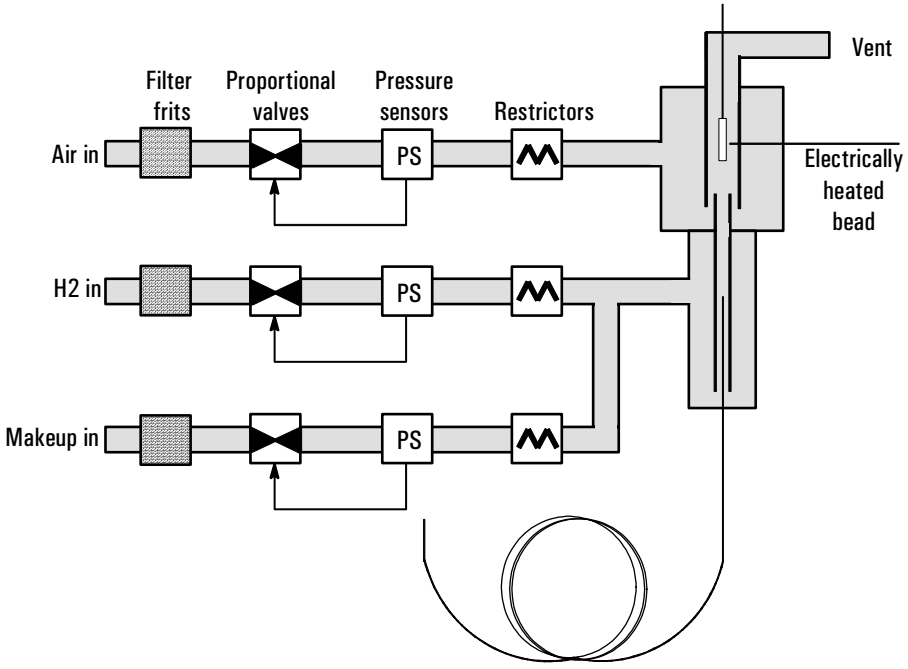


Figure 14. NPD pneumatics—EPC

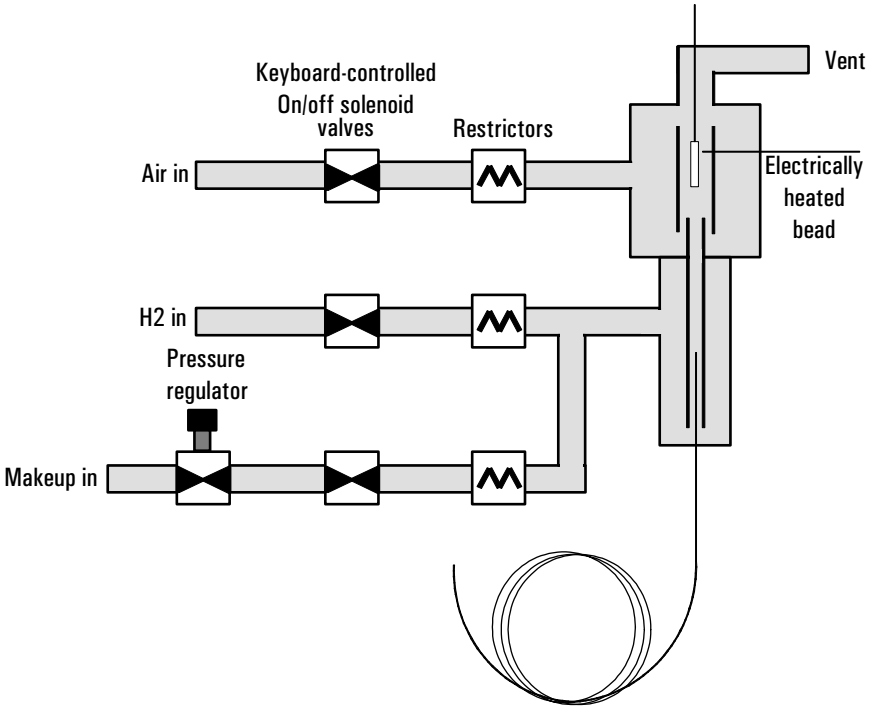


Figure 15. NPD pneumatics—NonEPC

Conditions that prevent the NPD from operating

- Hydrogen or air setpoints are set to 0.
- If the detector temperature is below 150°C or the oven is off, the `Adjust offset` process will not start.

Gas purity

Because of its high sensitivity, the NPD requires very pure gases. We strongly recommend that moisture and organics traps be used on the carrier gas and all detector gases, including the detector hydrogen, air, and makeup gases.

The bead

Two ceramic beads are available:

Bead color	Part no.	Advantages	Disadvantages
White	G1530-60570	Standard	Phosphorus tails
Black	5183-2007	Durable, no phosphorus tailing	Lower nitrogen sensitivity, about 40%

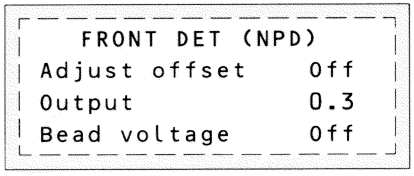
There are three setpoints associated with the bead—`Adjust offset`, `Bead voltage`, and `Equib time`.

Adjust offset

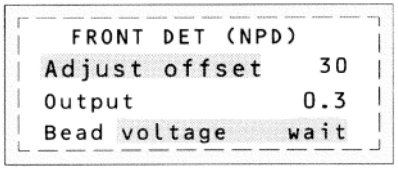
When you enter a value here, or press [On] to use the stored value, detector gas flows turn on, the bead heats, and the bead voltage adjusts until `Output` is stable and equal to the entered value. There are five stages of `Adjust offset`.

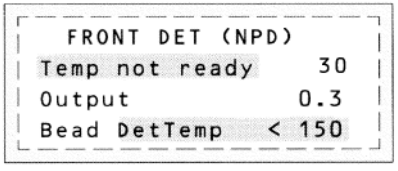
Detector off. When the detector is off, `Adjust offset` and `Bead voltage` are `Off` and initial `Output` is displayed.

Press [Front Det] [Det Control] or [Back Det] [Det control].

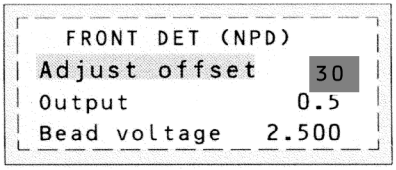


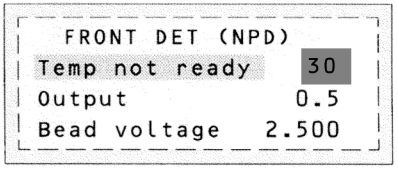
Detector on—detector temperature less than 150°C. When you enter an `Adjust offset` value or press [On], detector gases are off and the display blinks the following messages:



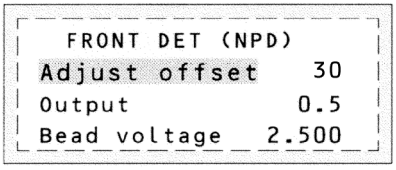


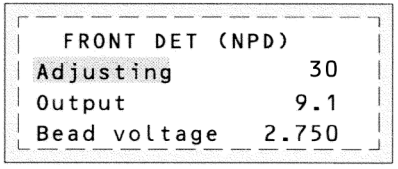
Detector on—waiting for oven and/or detector to reach temperature setpoint and equilibrium. When the detector temperature exceeds 150°C, the hydrogen and air flows turn on and the bead begins to heat while the oven and detector reach setpoint and equilibrate. The display blinks:



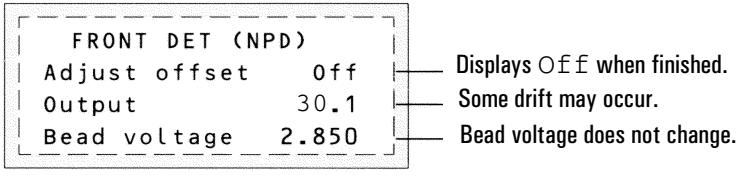


Detector on—during adjust offset and equilibration time. When the detector and oven temperatures reach setpoint and equilibrate, the `Adjust offset` process begins. The bead voltage is increased until the output is close to the `Adjust offset` value. Equilibration time (see the next page) begins. The display blinks.





Detector on and ready. When the `Adjust offset` value is reached and the equilibration time has passed, the `Adjust offset` line is `Off`. Your detector is on and ready.



Aborting adjust offset

Press [Delete] with the cursor on the `Adjust offset` line. This cancels the adjustment without turning off the detector gases and bead voltage. This is useful if you wish to start a run before the bead equilibration time is passed.

Turning off the detector

Caution If you turn `Adjust offset` [Off] at any time, the bead voltage, hydrogen, and air flows all turn off.

Setting adjust offset on the clock table

You can use the Clock table feature to turn the `Adjust offset` on at a specified time. Details can be found in chapter 9, "Instrument Automation", in the *General Information* volume.

Caution It is not recommended that you `Adjust offset` between runs. Before the oven reaches its initial setpoint and the system is thermally stable, column bleed and residual peak tailing can mask an otherwise stable baseline. This can waste time between runs.

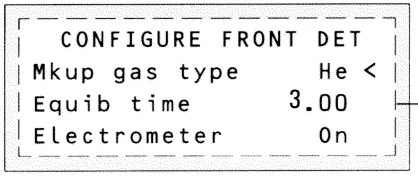
Equilibration time

Equilibration time begins when `Output` nears the `Adjust offset` value. During equilibration, `Output` is measured and compared to the `Adjust offset` value. If `Output` stays close to the `Adjust offset` for the entire equilibration time, the detector becomes ready. However, if the `Output` is too high or too low at any time during the equilibration period, the adjust offset process continues and the equilibration time begins again

We recommend an equilibration time of 0.0 minutes and the automatic `Adjust offset` process. Some beads do not respond well to the automatic process. For these, we suggest starting at 2.0 volts and bringing up the bead voltage gradually, 10 mV at a time, until the desired offset is reached.

Procedure: Changing equilibration time

1. Press [Config][Front Det] or [Config][Back Det]:



2. Enter a value (in minutes). Long equilibration time reduce bead lifetime.

Turning hydrogen off during a solvent peak

When using the NPD, the baseline shifts after a solvent peak and can take some time to stabilize, especially with chlorinated solvents. To minimize this effect, turn off the hydrogen flow during the solvent peak and turn it back on after the solvent elutes. With this technique, the baseline recovers to its original value in less than 30 seconds. This also extends the life of the bead. The hydrogen can be turned on and off automatically as part of a Run Table. See chapter 7, “Instrument Automation”, in the *General Information* volume.

Turning hydrogen off between runs

To extend bead life, turn off the hydrogen flow between runs. Leave all other flows and the detector temperature on. Turn on the hydrogen flow for the next run; the bead will ignite almost immediately. The process can be automated with Run Table entries.

Bead voltage

`Bead voltage` shows the voltage used to heat the bead. It can be an actual value, dependent on the `Adjust offset` value, or can be entered as a setpoint.

Equilibration time is not used when you enter a setpoint for `Bead voltage`, so you cannot estimate your baseline stability. Use the `Bead voltage` setpoint when the automatic startup does not work.

`Bead voltage` is also useful for small adjustments between runs. If you observe a baseline drift, you can enter a small, one-time change to compensate for the drift without having to wait for the `Equib time`.

Typical voltages for new beads range from 2.5 to 3.7 volts. Higher values reduce bead life.

Extending the life of the bead

- Use the lowest practical adjust offset or bead voltage.
- Run clean samples.
- Turn the bead off when not in use.
- Keep the detector temperature high (320 to 335°C).
- Turn the hydrogen flow off during solvent peaks and between runs.
- If your NPD is `Off` for a long time in a high-humidity environment, water may accumulate in your detector. To evaporate this water:
  a. Set the detector temperature at 100°C and maintain it for 30 minutes.
  b. Set the detector temperature to 150°C and maintain it for another 30 minutes.

Temperature programming

The NPD is flow sensitive. If you are using temperature programming, in which the column flow resistance changes with temperature, set up the instrument as follows:

- With an EPC inlet, set the carrier gas in the `Constant flow` mode. Set EPC detector makeup gas to `Const makeup`. NonEPC detectors provide constant makeup gas flow.
- If you have an EPC inlet and an EPC detector and choose to work in the constant pressure mode, the makeup gas should be set in the `Col+mkup=const mode`.

Electrometer

The Configure Detector control table contains an `On/Off` setpoint for the `Electrometer`. You do not need to turn the electrometer on and off when operating your NPD.

Caution Do not turn off the electrometer during a run. It will turn off the detector `Output`.

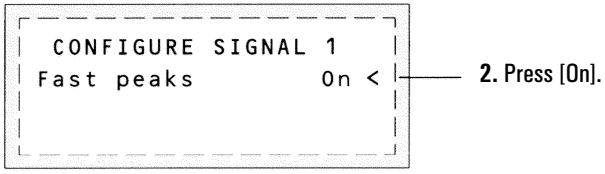
Data rates

Analog output for the NPD can be presented at either of two speeds. The faster speed allows minimum peak widths of 0.004 minutes, while the standard speed allows peak widths of 0.01 minutes.

Procedure: Setting data rate for NPD

If you are using the *fast peaks* feature, your integrator must be fast enough to process data coming from the GC. Integrator bandwidth be at least 15 Hz. To use fast peaks:

1. Press [Config][Signal 1] or [Config][Signal 2].



Digital output to the ChemStation is available at eleven speeds ranging from 0.1 Hz to 200 Hz, capable of handling peaks from 0.001 to 2 minutes wide. Output to an INET integrator is available at 20 Hz. Consult “Signal Handling” in the *General Information* volume for a discussion of the different rates.

The *fast peaks* feature does not apply to digital output.

Jets and collectors

The *capillary optimized* NPD is only used with capillary columns. It is shipped with the standard jet and collector.

Table 5. Jets and Collectors for the Capillary-Optimized NPD

Type	Part no.	id	Use with
Standard jet	G1531-80560	0.29 mm	Either collector
Extended jet (optional)	G1534-80580		Either collector
Standard collector	G1534-20530	7 mm	
Small id collector (optional)	G1534-20660	5 mm	

The *adaptable* NPD fits packed columns and can be adapted to fit capillary columns. It is shipped with the capillary column jet and standard collector. You must change the jet to use packed columns. Instructions appear later in this chapter.

Table 6. Jets and Collectors for the Adaptable NPD

Type	Part no.	id	Use with
Capillary column jet	19244-80560	0.29 mm	Either collector
Extended jet	G1534-80590		Either collector
Standard collector	G1534-20530	7 mm	
Small id collector	G1534-20660	5 mm	

The extended jets, used with the small id collectors, reduce the exposure of the sample to heated metal and reduce tailing of some very polar components.

Part 2. Operating the NPD

Use the information in Table 7 to select temperatures and flows. Choose a minimum source pressure from Figure 16. If you have an EPC detector, you must add 10 psi (69 kPa) to the source pressure on the chart.

Table 7. Flows, Temperatures, and Bead Information

Gas type	Recommended flow
Carrier gas *(helium, hydrogen*, nitrogen)*	*Capillary*, choose optimum flow based on column dimensions.
Detector gases	
Hydrogen	3.0 mL/min (maximum flow is 5 mL/min)
Air	60 mL/min
Capillary makeup *(helium,** nitrogen)*	Nitrogen: 5 to 10 mL/min Helium: less than 5 mL/min
Temperature (Default is 250°C; range is ambient to 400°C) $<150^{\circ}\times$C, the `Adjust offset` process will not start. 325 to 335°C is recommended. Detector temperature should be greater than the highest oven ramp temperature. With higher detector temperatures, less bead heating voltage is required.	
Adjust offset Default is 30 pA, suggested operating range is 30 to 40 pA, and allowable range is 10 to 99 pA. ≥50 pA increases sensitivity but reduces bead life. Lower settings reduce sensitivity and increase bead life, but too low will result in solvent quenching. Once Adjust offset is turned on, allow 20 to 60 minutes for detector to reach readiness.	
Equib time (Default is 5 minutes; range is 0 to 999.9 minutes) Recommended time is 0.0 minutes.	
Bead voltage (range is 0 to 4.095 V) Use to make minor adjustments or manually activate the bead. Set Equib time = 0.0 and Bead voltage at 2.0. Increase voltage in 0.01 volt increments until the bead ignites.	

Gas pressures

EPC detector—choose a flow, find a pressure, set source pressure 10 psi higher.
Non-EPC detector—choose a flow, use charts to find a starting source pressure.

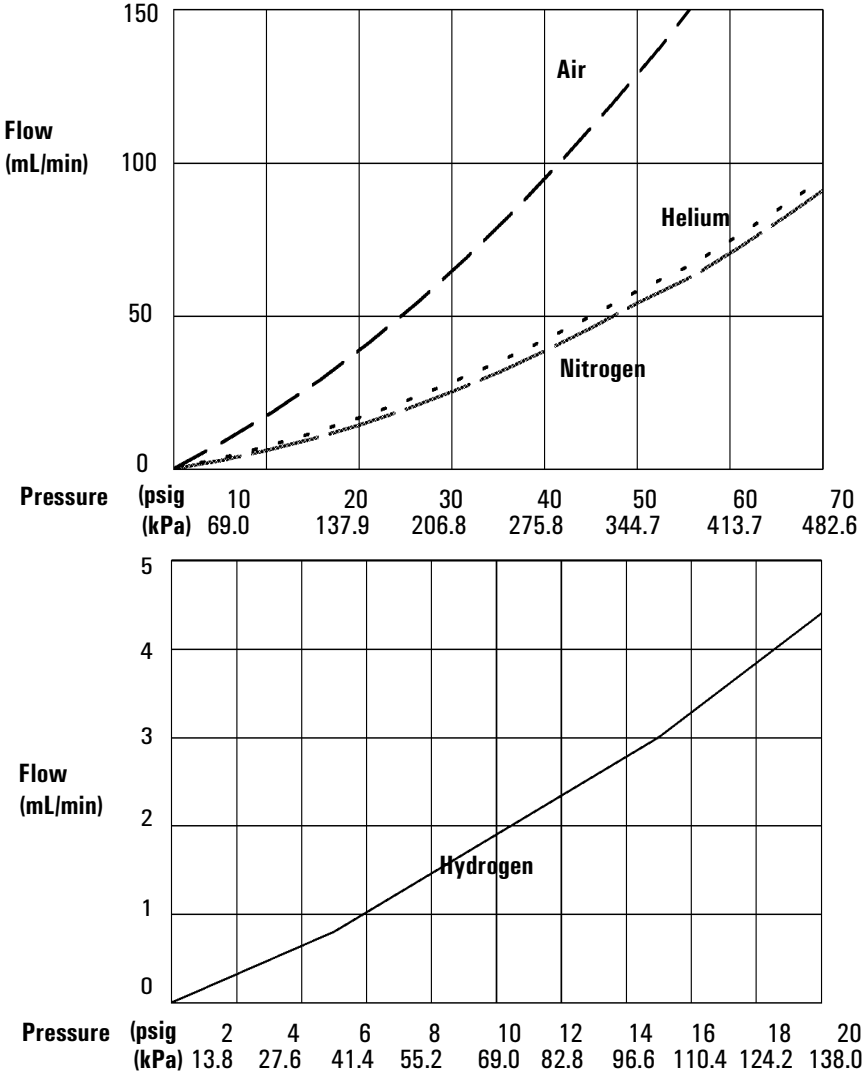
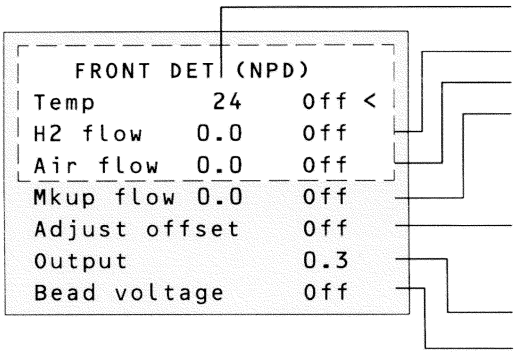


Figure 16. Pressure/flow relationships for NPD gases
(at 25°C and 1 atmosphere of pressure)

Operating with EPC

Press [Front Det] or [Back Det].



Temperature °C

Hydrogen flow, mL/min

Air flow, mL/min

Turn off for packed columns.
For capillary columns, see **makeup gas flow mode** below.

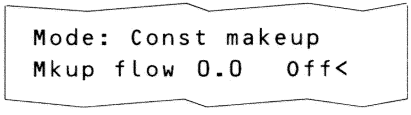
Adjust bead voltage automatically to achieve stable Output.
(10 to 99 pA).

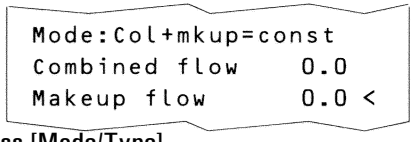
Actual value of detector output, pA

Actual bead heating voltage
(0 to 4.095 V)

Makeup gas flow mode

If column dimensions are specified and you have an EPC inlet, the control table will also include one of these:

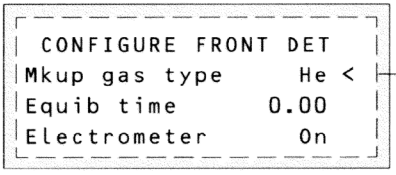




To **change makeup mode,** scroll to Mode: and press [Mode/Type].

Make a selection and enter the appropriate flow values.

To change **makeup gas type** or **equilibration time,** press [Config][Front Det] or [Config][Back Det]:



You do not need to turn the electrometer on or off.

Press [Mode/Type] to **change makeup gas:**

Select the appropriate gas.

Figure 17. NPD control table with EPC

Procedure: Using the NPD with EPC

Before operating the NPD, make sure that detector gases are connected, a column is installed, the correct jet is installed, and the system is free of leaks. Set the oven temperature, inlet temperature, and column flow. Use the information in Figure 17 when editing the control tables.

WARNING Make sure that a column is installed or the NPD column fitting is plugged before turning on the air or hydrogen. An explosion may occur if air and hydrogen are allowed to leak into the oven.

1. Press [Config][Front Det] or [Config][Back Det].
   a. If you are using makeup gas, verify that the configured makeup gas type is the same as that plumbed to your instrument. Change the gas type, if necessary (page 7). Nitrogen is recommended.
   b. Check the equilibration time. The recommended value is 0.0.
2. Press [Front Det] or [Back Det] to open the NPD control table.
3. Set the detector temperature. The recommended range is 325 to 335°C.
4. Enter a hydrogen flow (3.0 is recommended), if desired, and press [Off].
5. Enter an air flow (60 is recommended), if desired, and press [Off].

   If you are using ***packed columns***, turn off makeup gas and proceed to step 7.

   If your ***capillary column*** is *defined* and connected to an EPC inlet, choose a new flow mode (page 7), if desired, and set the makeup gas flow. If you have set up your column in the constant flow mode, choose `const makeup`. If you have set up your column in the constant pressure mode, choose `Col+makeup=const`.

   If your column is *not defined*, or is connected to a nonEPC inlet, enter a makeup gas flow. Only constant flow is available.
6. Enter `Adjust offset` number, or press [On] to begin the adjustment process. Your hydrogen and air flows will be switched on once the detector reaches 150°C.

Short-cut procedure:
(assumes correct setpoints are stored)

1. Open detector control table.
2. Turn temperature On.
3. Turn makeup gas On, if needed.
4. Press [Det Control].
5. Press [On].

Operating without EPC

Press [Front Det] or {Back Det].

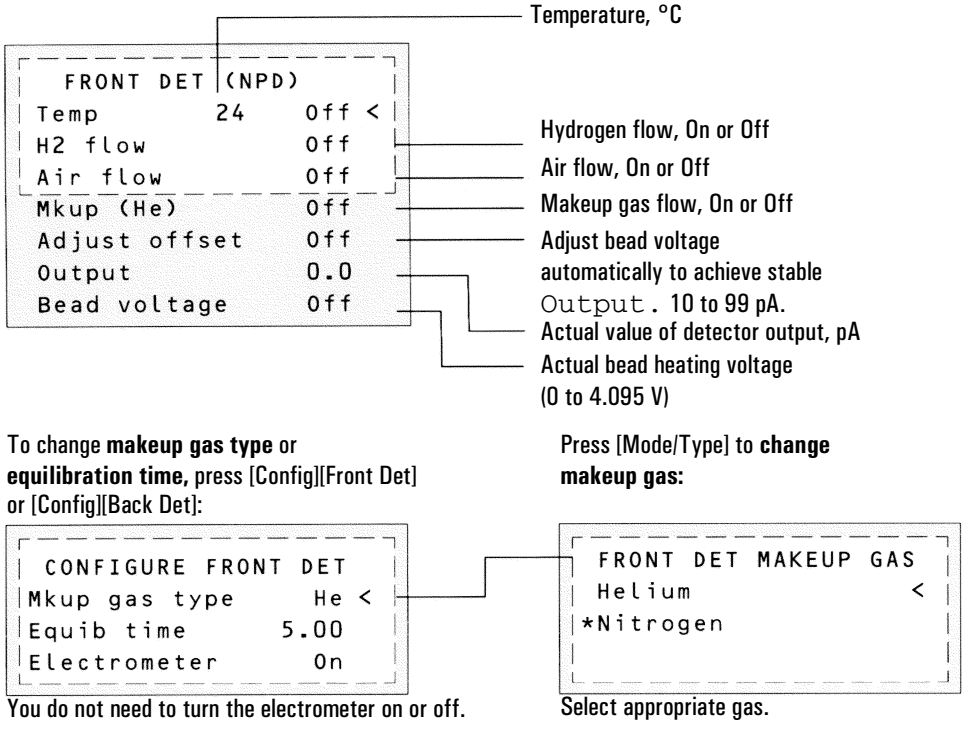


Figure 18. NPD control table without EPC

Procedure: Using the NPD without EPC

Verify that detector gases are connected, a column is installed, the correct jet is installed, and the system is leak-free. Set oven temperature, inlet temperature, and column flow.

Use a bubble meter with an NPD adapter (part no. G1534-60640) to measure flows. Remove the bead and push the adapter into the collector. You are now ready to measure the flow. Quicker measurements can be made by measuring at the detector vent. This measurement is ≈95% accurate when total flows exceed 50 mL/min.

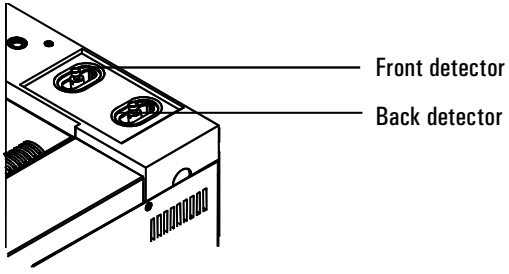
1. Press [Config][Front Det] or [Config][Back Det].
   a. If using makeup gas, verify that gas type is the same as that plumbed to your instrument. Change the gas type, if needed (page 7).
   b. Check the equilibration time. Enter a new number, if desired.
2. Press [Front Det] or [Back Det].

WARNING To minimize the risk of explosion, never measure air and hydrogen together. Measure them separately.
Make sure a column is installed or the NPD column fitting is plugged before turning on the hydrogen. An explosion may occur if hydrogen leaks into the oven.

3. Adjust the hydrogen flow.
   a. Make certain the air and makeup gas are turned off.
   b. Turn the hydrogen flow on.
   c. Set the supply pressure and measure the resulting flow.
   d. Adjust supply pressure until the hydrogen flow is correct. If you have flow through your column, subtract it from the total flow.
   e. Turn off the hydrogen from the control table while you measure air flow.

4. Adjust the air flow.
   a. Make sure the hydrogen and makeup gas are turned off.
   b. Turn the air flow on.
   c. Set the supply pressure and measure the resulting flow.
   d. Adjust the supply pressure until the air flow is correct. If you have flow going through your column, subtract it from the total flow.
   e. Turn the air flow off.
5. If you are using ***packed columns***, turn the makeup gas off and proceed to step 7.
6. If you are using ***capillary columns***, set the makeup gas flow. Make sure the hydrogen and air flows are off while you measure the flow.
   a. Set the supply pressure.
   b. Locate the knob that controls the pressure regulator on the flow manifold. Turn the knob *clockwise* to increase flow and *counterclockwise* to decrease flow.

Makeup gas pressure regulator



   c. Measure the flow using a flow meter.
   d. Adjust the regulator until the makeup flow is correct. If you have flow going through your column, subtract it from the total flow.
7. Enter `Adjust offset` number, or press [On] to begin the adjustment process. The hydrogen and air flows are switched automatically on once the detector temperature exceeds 150°C.

Short-cut procedure:
(assumes correct flows have been set and setpoints are stored)

1. Open detector control table.
2. Turn temperature On.
3. Turn makeup gas On, if needed.
4. Press [Det Control].
5. Press [On].

Part 3. Checkout Conditions and Chromatogram

This section contains a typical example of a test sample chromatogram. It may be used as a general guide to instrument performance.

Note that injection volumes listed with operating conditions do not necessarily indicate total absolute volume injected. Volume given is simply the graduation (plunger position) read from a standard 10 µL syringe. For a heated inlet, actual sample volume injected will also include an additional 0.4-0.7 µL, the volume of sample volatilized from inside the syringe needle. For the dedicated, on-column inlet (unheated), the syringe plunger position more accurately reflects the true injected volume.

Also note that the following procedure and results are intended only to provide evidence of a properly functioning inlet and/or detector system; they are not necessarily suitable to test a given system against its specification limits.

NPD checkout conditions

Column and sample

Type	5 30m × 0.32mm × 0.25 μm PN 19091J-413
Sample	NPD Checkout 18789-60060
Injection volume	1 μL

Inlet

Temperature	200°C Purged/Packed or Split/Splitless
	Oven Track Cool On-Column
	60°C PTV (see below)
Inlet pressure	25 psi (Constant pressure for EPC inlets, helium)
Split/Splitless	
Mode	Splitless
Purge flow	60 mL/min
Purge time	0.75 min

Inlet, continued

PTV

Mode	Splitless
Inlet temperature	60°C
Initial time	0.1 min
Rate 1	720°C/min
Final temp 1	350°C
Final time 1	2 min
Rate 2	100°C/min
Final temp 2	250°C
Final time 2	0 min
Inlet pressure	25 psi (Constant pressure for EPC inlets)
Purge time	0.75 min
Purge flow	60 mL/min

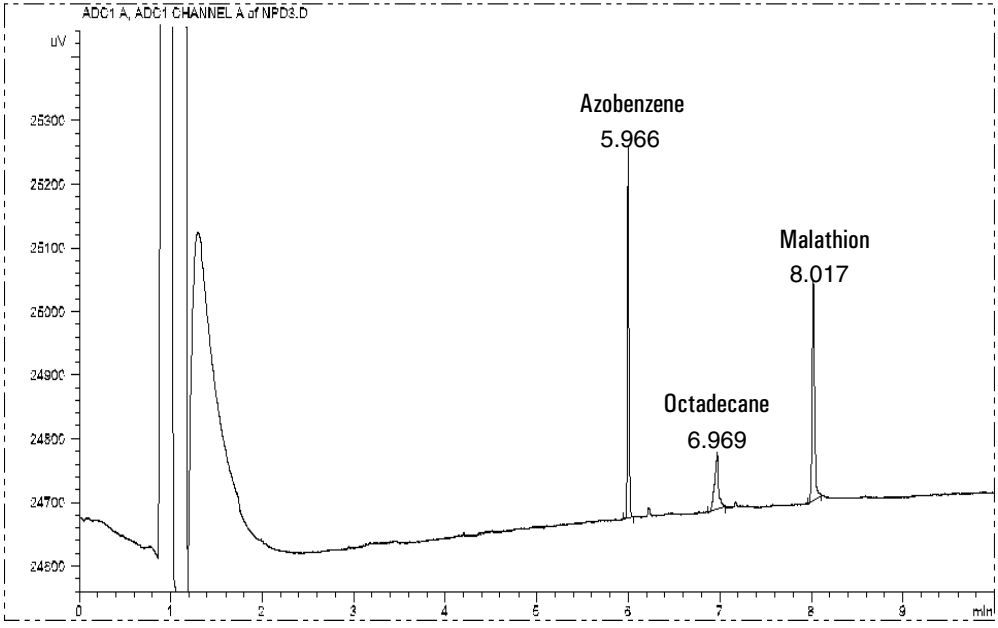
Detector

Temperature	300°C (325 to 330°C recommended)
H_2 flow	3 mL/min
Air flow	60 mL/min
Makeup + column flow	10 mL/min (nitrogen recommended)
Offset	50 pA (30 to 35 recommended)

Oven

Initial temp	60°C
Initial time	0 min
Rate 1	20°C/min
Final temp	200°C
Final time	3 min

NPD checkout chromatogram



Your retention times will differ but peaks should resemble the example.

Part 4. Maintaining a Nitrogen-Phosphorus Detector

NPD illustrated parts breakdown

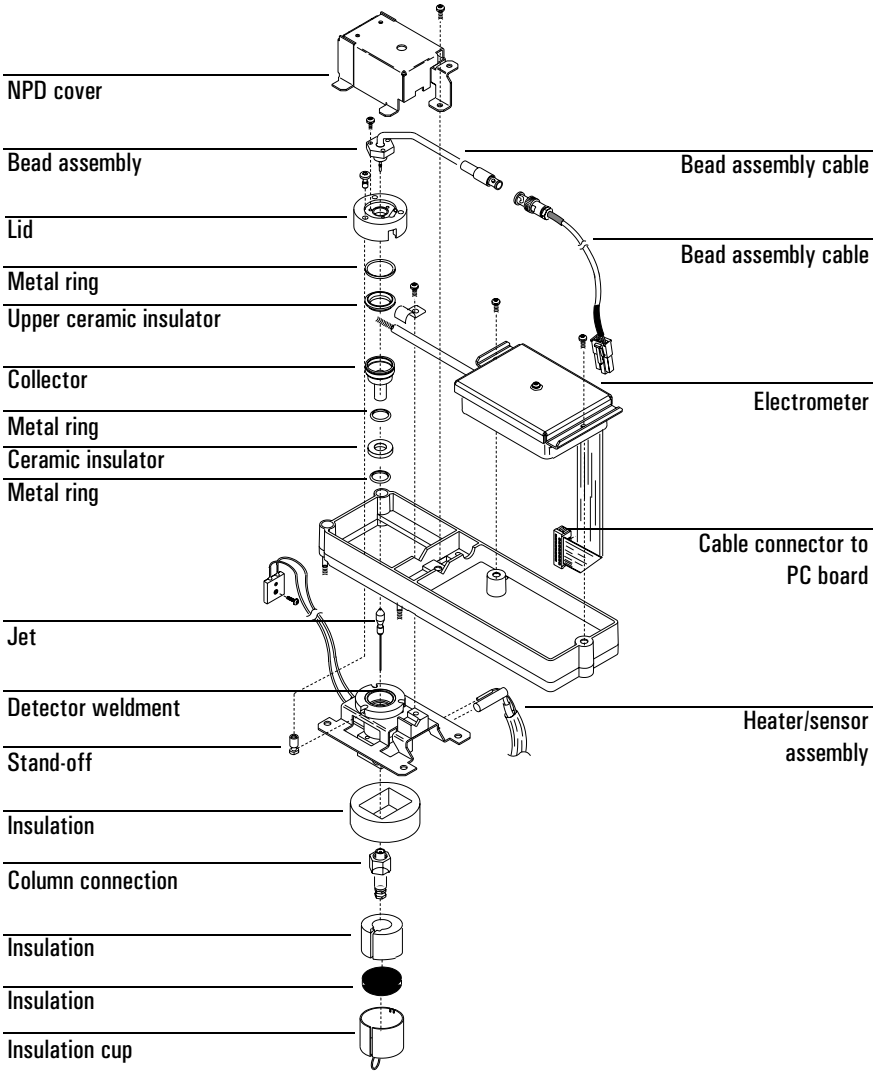


Figure 19. The NPD

Correcting NPD hardware problems

No detector response to injected sample

- A large concentration of solvent has extinguished the hydrogen/air plasma. Increase the bead voltage. Run the detector at a higher offset (for example, 40 to 50 pA), or use makeup gas at a flow rate of 5 mL/min.
- Check that hydrogen is flowing to the detector. Verify that there is hydrogen coming from the external supply. Check that flow and pressure are turned on at the keyboard. The hydrogen flow rate should be between 1.0 and 5.5 mL/min.
- The bead is not activated. Look through the vent hole on the detector lid to see if the bead is glowing orange. If the bead is not glowing, check that there is enough current reaching the bead. Check the detector background signal. Reduce the bead voltage to zero to establish a reference level, and then look for a sudden sharp increase in output as the bead voltage increases, which indicates that ignition occurred. If 4 V are being supplied to the bead but it is not igniting, the bead is probably burned out. Replace the bead.
- The bead power cable is bad. Contact your Agilent service representative.
- If the upper ceramic insulator is contaminated, a high offset (2 to 15 pA or more) will occur when the bead is off. This directly affects sensitivity. Replace the ceramic insulator.

No baseline; output signal exceeds 8 million

- The electrometer ribbon cable is not attached to the PC board properly. Be sure to turn the GC off before reattaching the cable! If the signal does not drop to a normal level (<3 pA), you need to replace the electrometer. Contact your Agilent service representative.
- The collector is shorted to the detector housing. Check the insulators.

Baseline level is 0.0

- Broken electrometer. Contact your Agilent service representative.

Large positive baseline upset with very slow recovery to original baseline

- The solvent contains significant concentrations of chlorinated hydrocarbon. Create a time table that turns hydrogen off at the time of injection. When the solvent has passed through the detector, restore the hydrogen flow to the previous operating level. The NPD will usually recover rapidly to a stable baseline.

Baseline does not recover after solvent peak

- Create a time table that turns hydrogen off at the time of injection. When the solvent has passed through the detector, restore the hydrogen flow to the previous operating level. The NPD will usually recover rapidly to a stable baseline.

  Add makeup gas at a flow rate of 5 mL/min.

  A large concentration of solvent has extinguished the hydrogen/air plasma. Increase the bead voltage. Run the detector at a higher offset (for example, 40 to 50 pA).

Adjust offset does not function properly (it is either too high or too low by hundreds of pA)

- A flame is burning at the top of the jet. If the hydrogen flow is too high, the flame at the tip of the jet will continue burning. Turn off the hydrogen flow completely, and decrease the flow rate. The hydrogen flow should never be higher than 4.0 mL/min.

Large solvent signal with very small NPD signal

- Check the hydrogen flow rate. If it is too high, a flame could be burning at the tip of the jet. Turn off the hydrogen flow completely, and decrease the flow rate. The hydrogen flow should never be higher than 4.0 mL/min.
- The collector may be contaminated. Change the collector and ceramic insulators.

Peak tailing

- Verify that a good liner and column are being used.
- Some polar compounds tail due to contact with the metal collector. The optional extended jets are recommended.
- Some compounds cause peak tailing, especially those containing phosphorus. The optional black ceramic bead is recommended for phosphorus.

The baseline drifts (upward) significantly during an oven program

- If the oven temperature is increasing dramatically during a run (for example, from 50 to 350°C) a change of between 10 and 15 pA is normal. However, if you suspect that the baseline drift is excessive, heat the inlets and oven to a temperature above 300°C for 60 minutes to eliminate excess baseline drift during oven programs.
- Verify that the detector insulation is not cracked or damaged.

High detector baseline of GC at room temperature

- Moisture in the detector can cause the baseline to be at 10s or even 100s of pA when the detector is at a low (such as room) temperature. Set the detector temperature to 150°C with the detector gases on. The baseline should drop below 1 pA in approximately 10 minutes.

The signal baseline does not fall below 3 pA when the bead voltage is 0

- The ceramic insulators may be dirty. The insulators must be very clean for NPD performance to be satisfactory. Refer to the cleaning procedure on page 99, “Cleaning collector and detector, changing insulators and rings.”

Procedure: Replacing the bead assembly

The bead, which is also referred to as the “source,” is the active part of the NPD. The bead is part of an assembly consisting of a cable that terminates in a connector and a metal hex on which the ceramic bead is mounted. The NPD

bead assembly needs to be removed for replacement or to allow you to access the collector for cleaning.

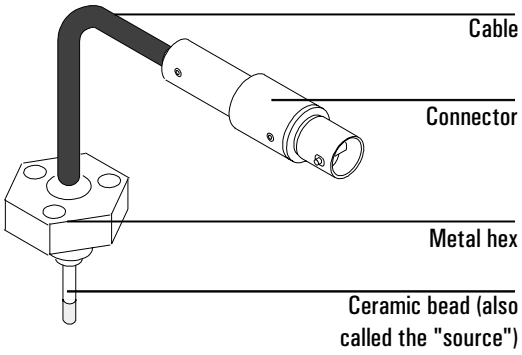


Figure 20. The NPD bead assembly

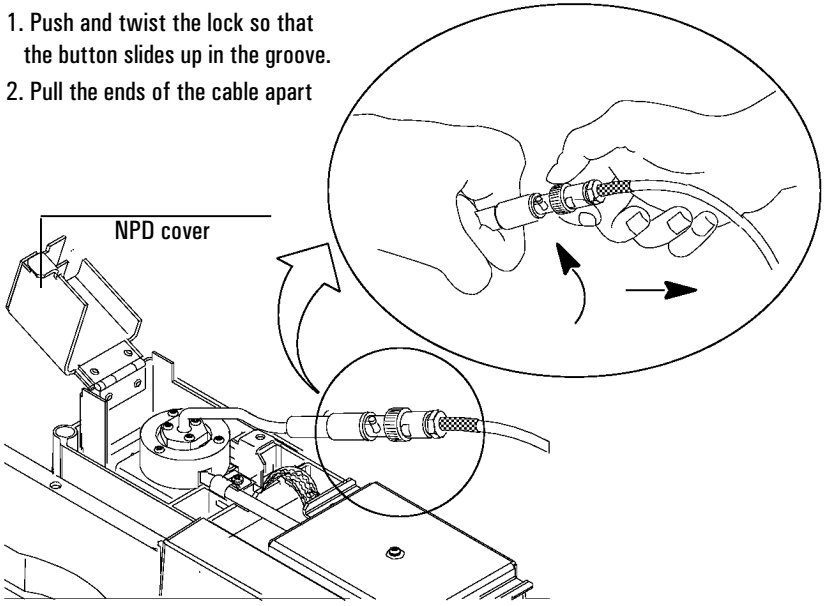
Caution The ceramic bead is delicate. Be careful not to break or crack the bead. When you perform maintenance on the NPD, avoid touching the bead with your fingers, and prevent it from coming in contact with other surfaces.

Caution Be careful! The oven or detector fittings may be hot enough to cause burns.

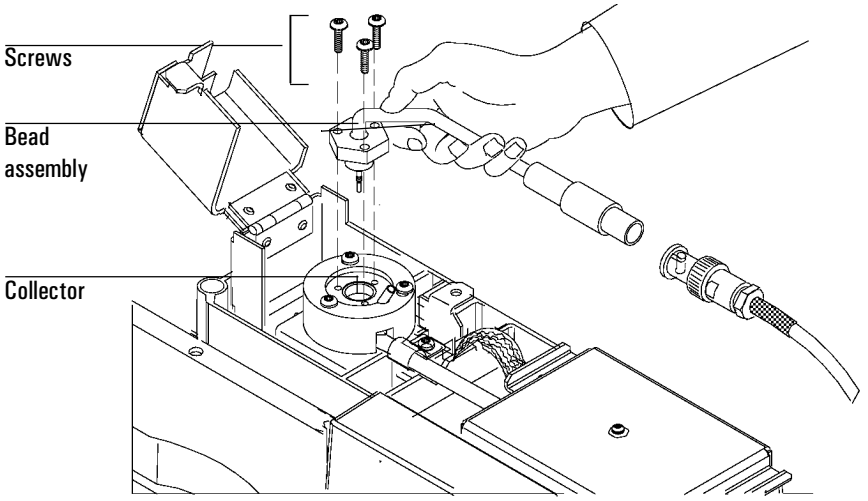
Materials needed:

- T-10 Torx screwdriver
- Cap for the bead

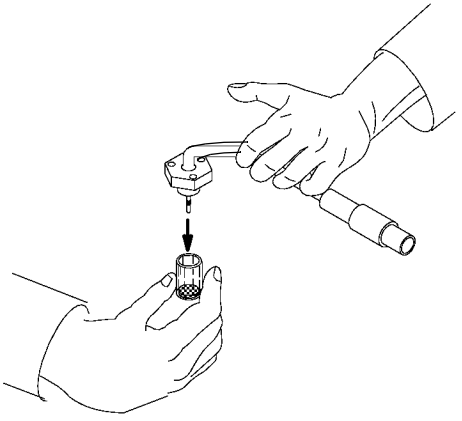
1. Complete the following preliminary steps:
   - Cool the detector to 100°C or lower before changing the bead.
   - Raise the GC top cover and open the NPD cover to cool the detector faster.
   - Turn the detector off. Set the bead voltage to less than 2.0 volts. Leave all gases on.
   - Remove the GC detector top cover and remove the electronics top cover.
2. Disconnect the cable by twisting the ring and pulling the ends apart.



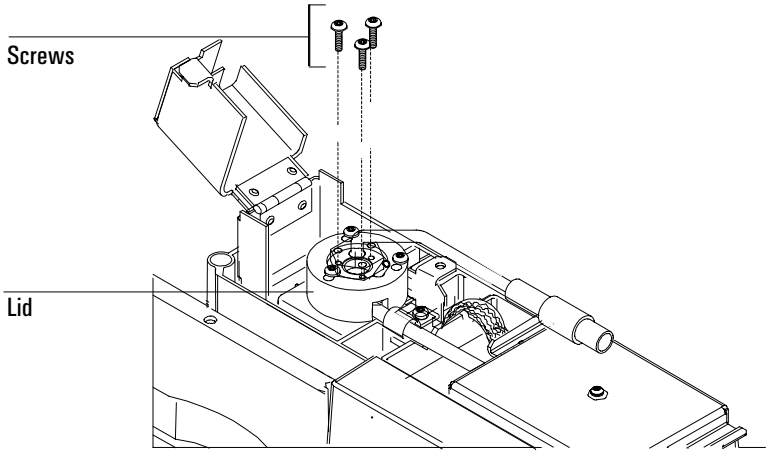
3. Use the Torx screwdriver to remove the three screws on the bead assembly. Grasp the cable gently and lift the bead assembly straight up. Avoid bumping the bead against the sides of the collector.



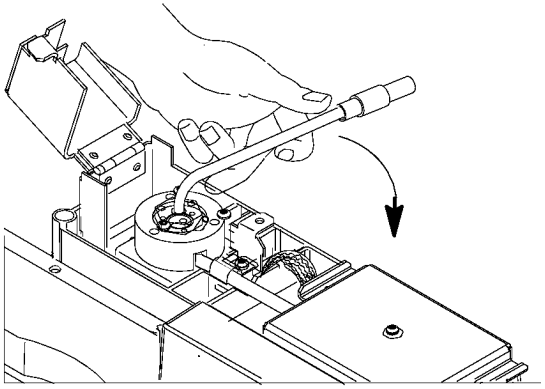
4. Uncap the new bead by pushing the cap off from the cable side. Make sure not to bump the bead on the sides of the cap.



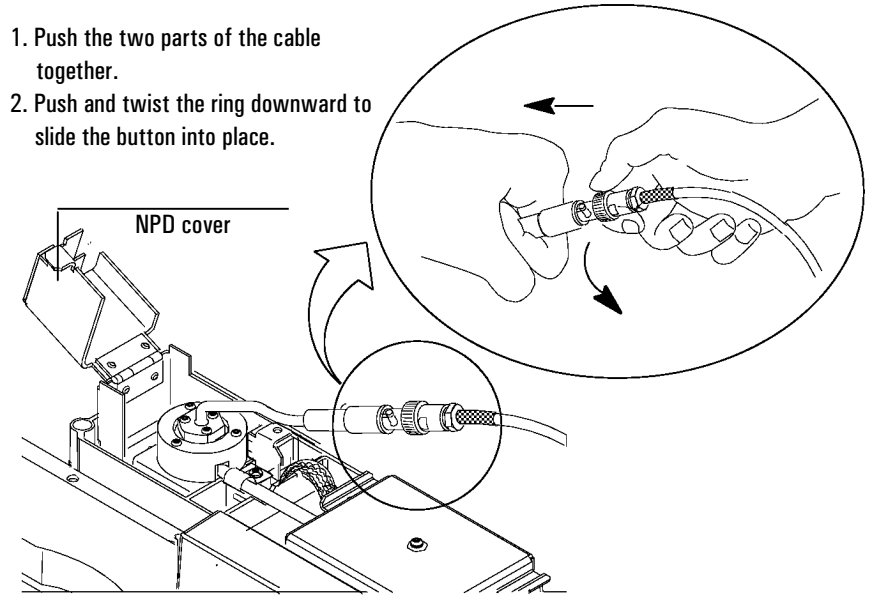
5. Mount the new bead assembly on the NPD lid. Be careful not to bump the bead on the sides of the lid or collector. Replace the three screws. Tighten the first screw only finger-tight; tighten the remaining two screws normally and then completely tighten the first screw.



6. Carefully bend the bead assembly cable 90 degrees. You should support the bead as shown below.



7. Reattach the bead assembly power cable to the NPD power cable and twist the ring to lock the connection.



8. Close the NPD cover and the GC detector cover. Replace the electronics top cover. You must close all three covers to get a stable NPD baseline. You can also restore normal operating conditions.

9. Heat the detector to 150°C for about 15 minutes. Then increase the temperature to the operating value (325 to 335°C recommended). Allow 15 minutes for equilibration.

10. Set Equilibration time to 0.0. Either start `Adjust offset` or gradually raise the bead voltage, about 0.01 volts at a time, until the baseline increases to the desired offset.

Procedure: Cleaning detector and collector; changing insulators and rings

Over time, residue from the bead or sample can build up in the collector and cause baseline problems. You should clean the collector after you have changed the bead two or three times.

The ceramic insulators must remain very clean to provide a steady baseline. Always wear gloves when handling the insulators. Clean insulators should provide no more than 1.0 pA, and usually about 0.5 pA, offset with the hydrogen turned off or the bead voltage at 0.

The metal C-rings wear a little with each assembly and disassembly. After several assemblies and disassemblies (five or more), the rings may not seal effectively, causing an erratic baseline. A ceramic insulator and seal kit is available (part no. 5182-9722). Always cool the detector to near-ambient when changing seals and insulators.

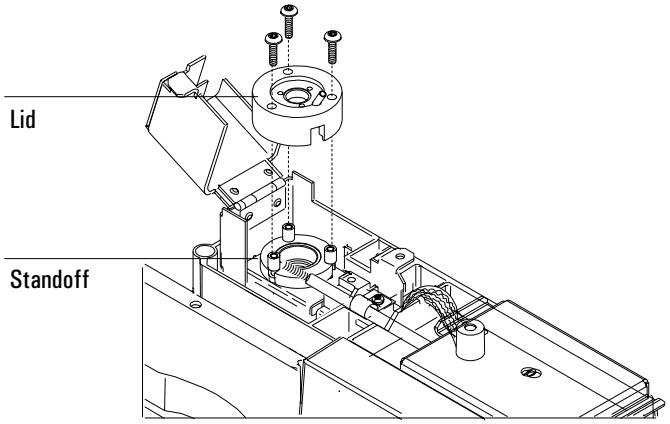
Caution Be careful! The oven or detector fittings may be hot enough to cause burns.

Materials needed:

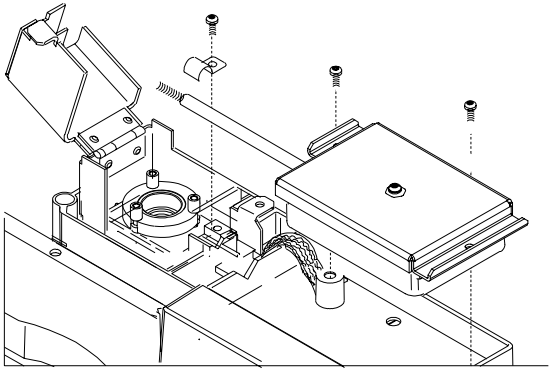
- T-10 and T-20 Torx screwdrivers
- Cap for the bead
- Cotton swabs
- Methanol or acetone
- Compressed air or nitrogen
- Lint-free gloves
- Forceps or tweezers
- New metal rings and ceramic insulators (kit part no. 5182-9722)

1. Complete the following preliminary steps:
   - Cool the detector to 60°C or lower. To cool the detector faster, raise the GC detector cover and open the hinged NPD cover.
   - Turn off the temperature, gases, and bead voltage.
   - Turn off the electrometer; press [Config] [Front Det] or [Config] [Back Det], scroll to `Electrometer` and press [Off].
   - Remove the electronics top cover.
2. Put on the lint-free gloves before touching any of the detector parts.
3. Remove the bead. Refer to the procedure on page 93 for instructions. Cap the bead carefully.

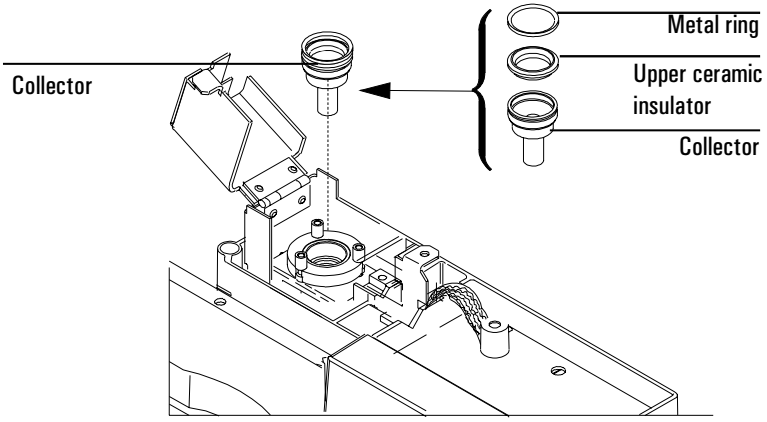
4. Using the T-20 screwdriver, remove the three screws that secure the lid, and then remove the lid. The metal ring and ceramic insulator may be attached to the lid.



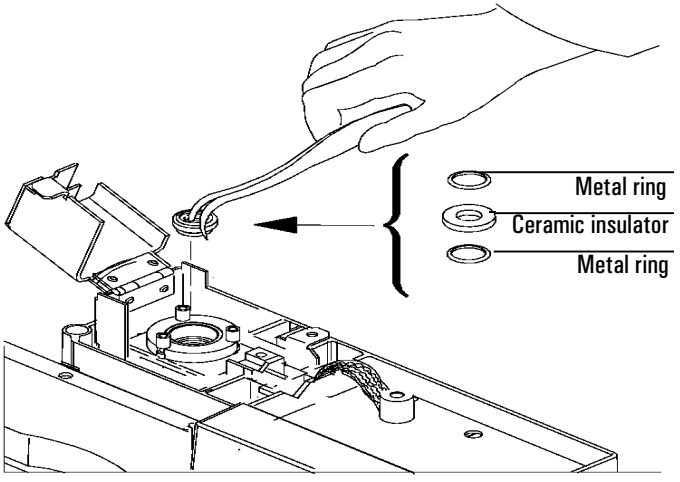
5. Remove the three screws that secure the electrometer and the interconnect. Pull the electrometer away from the detector to free the interconnect. Turn the electrometer to the right to obtain working space.



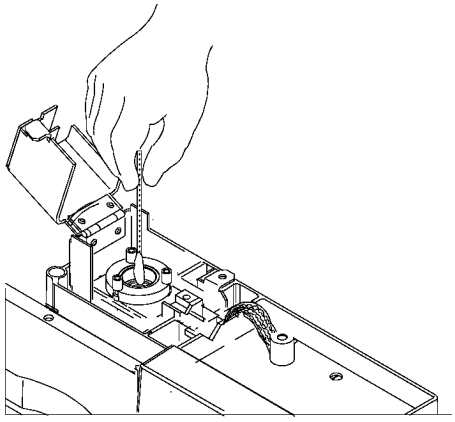
6. Remove the large metal ring and the upper ceramic insulator if they were not attached to the lid. Remove the collector. If you are operating the detector at high temperatures, these parts may stick inside the detector. Push and wiggle them to break the seal.



7. Using the forceps, remove the lower ceramic insulator and the two small metal rings located above and below it. If these parts are stuck together, do not separate them. If they are not stuck, remember which metal ring was on top of the insulator and which was below it! You will need to reassemble the pieces in the same orientation.



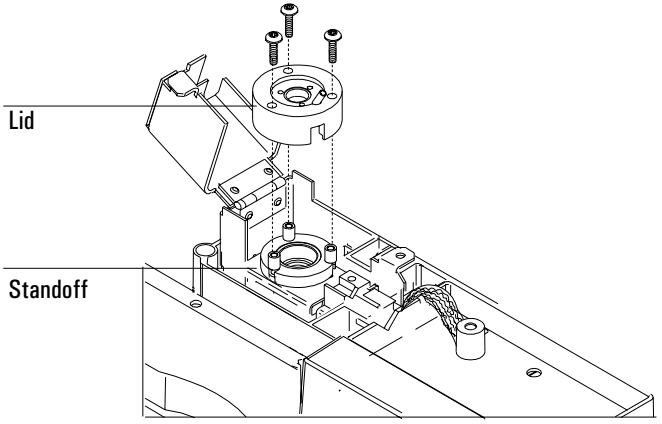
8. Use a cotton swab wetted with solvent to clean the residue from the inside of the collector and around the "cup." Also swab the detector base around the jet with a swab.



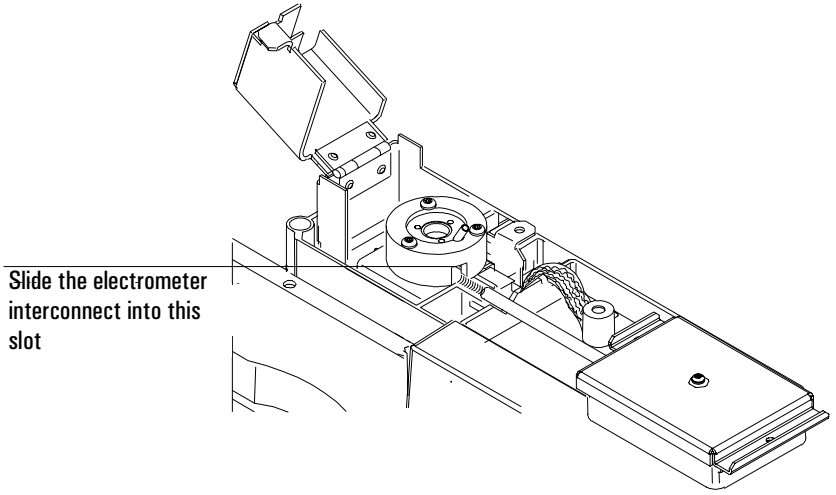
If the collector or the upper ceramic insulator are really dirty, cleaning may not help. Replace with new parts.

9. Reinstall the old or insert the new bottom metal ring, the lower ceramic insulator, and the second metal ring. Install the clean (or new) collector. Reinstall the old or insert the new upper ceramic insulator and large metal ring on top of the collector.

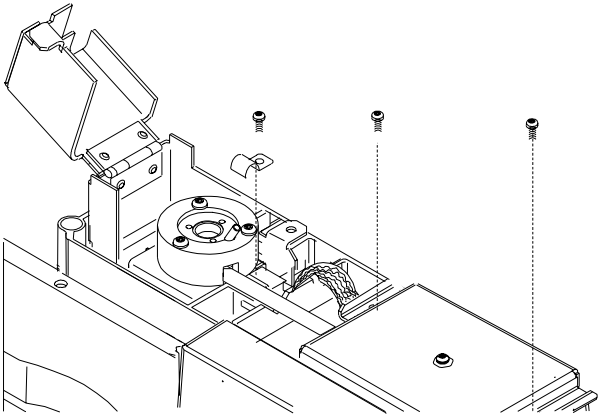
10. Replace the lid, making sure that the three standoffs are in their slots. Hold the lid flat while each of the three screws are tightened until they touch the lid. Tighten each one-half a turn at a time until tight.



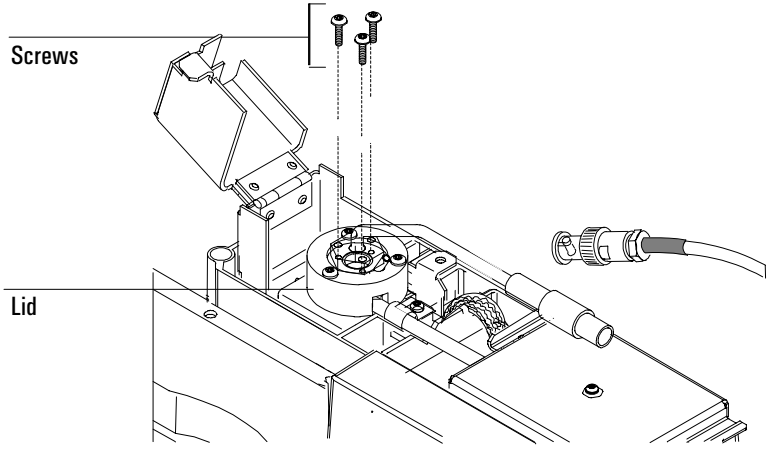
11. Slide the electrometer interconnect into the slot on the lid. Lower the electrometer into the mounting tray.



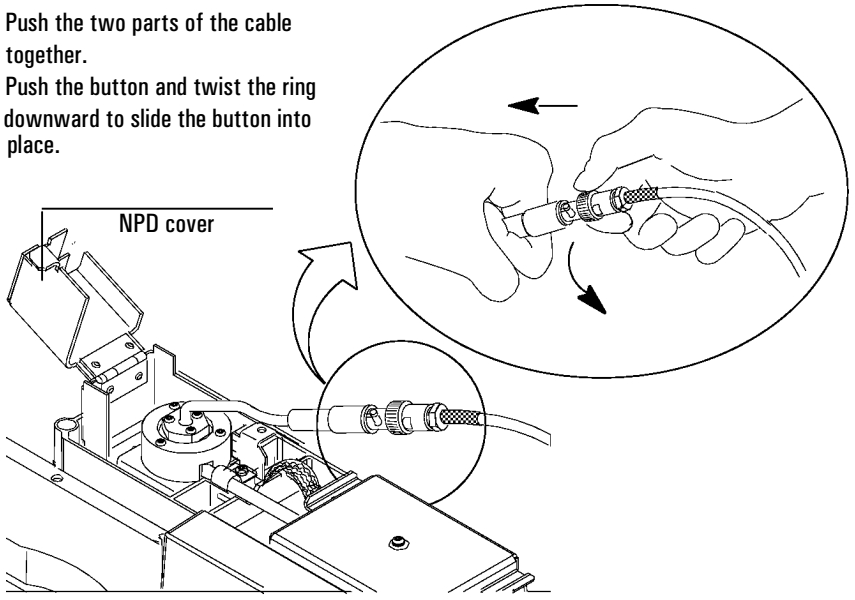
12. Replace the bracket, and replace and tighten the three screws.



13. Uncap the bead and replace the bead. Replace the three screws. Tighten the first screw only to snugness. Tighten the other screws completely, and then completely tighten the first screw.



14. Reattach the bead assembly cable to the NPD power cable and twist the ring to lock the connection. Close the NPD cover and the GC detector cover and replace the electronics top cover. You can restore normal operating conditions.



After reassembling the detector, you should check its operation. Turn on the gases, and then turn the bead voltage on to restore detector operation. Check that the offset reading is appropriate for your detector. If the values are not normal, the spring on the electrometer may not be contacting the detector correctly, there may be a leak at the column connection, or the detector may not have been reassembled correctly.

Replacing or cleaning the jet

Because there is no flame in the NPD, the jet does not collect silica and soot as does the FID jet. Although you can clean the jet, it is usually more practical to simply replace dirty jets with new ones. If you do clean the jet, use the cleaning wire (part no. 18765-20070), taking care not to damage the inside of the jet. You can also use a sonicator bath to clean the jet.

Table 8 lists the NPD jets.

Table 8. NPD Jets

Type	Part no.	Use with
Standard jet	G1531-80560	Capillary-optimized NPD
Extended jet (optional)	G1534-80580	Capillary-optimized NPD
Extended jet (optional)	G1534-80590	Adaptable NPD

There are four steps involved in cleaning the jet: removing the jet, inspecting it for damage or wear, cleaning the jet (optional), and replacing the jet and reassembling the detector.

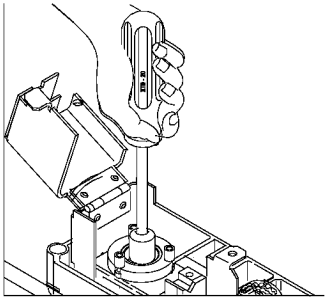
Procedure: Removing and inspecting the jet

Materials needed:

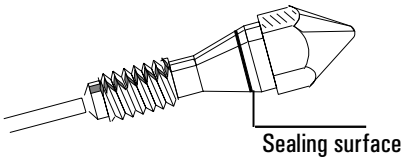
- T-10 and T-20 Torx screwdrivers
- 1/4-inch hex driver
- Cap for the bead
- Lint-free gloves
- Forceps or tweezers
- ESD wrist strap

1. Complete the following preliminary steps:
   - Raise the top cover and the NPD cover. Cool the detector to 60°C or lower. Turn off the inlet gases.
   - Turn off the temperature, gases, and bead voltage.
   - Turn off the electrometer; press [Config] [Front Det] or [Config] [Back Det], scroll to `Electrometer` and press [Off].
   - Cool the oven to room temperature. Remove the column from the detector end and cap the detector's column connection.
   - Open the GC detector cover and remove the electronics top cover.
2. Remove the collector, ceramic insulators and metal rings. Refer to the procedure starting on page 99.

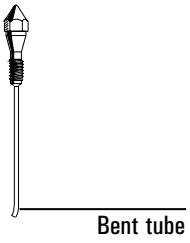
3. Using the nut driver, loosen the jet. Pull the jet straight out of the detector. You may need to use the forceps to remove it.



4. Inspect the jet sealing surface for scratches. You should see a small ring around the sealing surface; any other scratches, however, are unacceptable.



5. Inspect the jet tube to be sure it is not bent or crimped.



6. Inspect the jet for contamination by holding it up to a light and looking through its bore. If no contamination is present, the tube will be clear.

Caution The adaptable NPD extended jet is longer than the capillary-optimized NPD extended jet and should never be installed in a capillary-optimized detector.

Procedure: Cleaning the jet

It is often more convenient to replace dirty jets with new ones than to clean them, especially jets that have been badly contaminated.

Caution If you choose to clean a jet, be careful when using a cleaning wire. Be sure not to scratch the jet, because doing so will ruin it. You may want to skip the wire cleaning procedure and use the aqueous bath only.

Materials needed:

- Small ultrasonic cleaning bath
- Aqueous detergent
- GC-grade methanol in a Teflon wash bottle
- Flame detector cleaning kit (part no. 9301-0985)
- Dry, filtered, compressed air or nitrogen

1. Run a cleaning wire through the jet. Run it back and forth a few times until it moves smoothly. Be careful not to scratch the jet.
2. Aqueous cleaning procedure:
   a. Fill the ultrasonic cleaning bath with aqueous detergent, and place the jet in the bath. Sonicate for 5 minutes.
   b. Use a jet reamer to clean the inside of the jet.
   c. Sonicate again for 5 minutes.

   From this point on, handle the parts only with forceps!

   a. Remove the jet from the bath and rinse it thoroughly first with hot tap water and then with a small amount of methanol.
   b. Blow the jet dry with a burst of compressed air or nitrogen, and then place the jet on a paper towel to air dry.

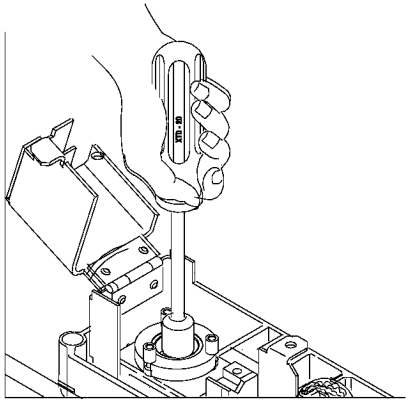
Procedure: Replacing the jet and reassembling the detector

Materials needed:

- T-10 and T-20 Torx screwdrivers
- Cap for the bead
- ESD wrist strap
- Lint-free gloves

Caution The adaptable NPD extended jet is longer than the capillary-optimized NPD extended jet and should never be installed in a capillary-optimized detector.

1. Place the jet in the detector body, and tighten it to snugness with the hex driver. Do not overtighten the jet.



2. Reassemble the detector. Refer to the procedure starting on page 103.

5

The Electron Capture Detector

Chapter 5
The Electron Capture Detector

Part 1. Regulatory and Safety Information

Agilent Technologies provides two types of electron capture detectors. The “regular” detector, abbreviated as ECD, has a larger internal volume (approximately 10 times) than the micro-cell detector, abbreviated as µ-ECD. The two types can be distinguished by the top cover of the detector—the ECD has a solid cover and the µ-ECD has a perforated cover.

This chapter describes the "regular" ECD. The micro-cell detector (µ-ECD) is described in the next chapter.

The ECD contains a cell plated with ^{63}Ni, a radioactive isotope. The ^{63}Ni releases β particles that collide with carrier gas molecules to produce low-energy electrons—each β particle produces approximately 100 electrons. The free electrons produce a small current—called the *reference* or *standing current*—that is collected and measured in a pulsed circuit.

When a sample component molecule comes into contact with the free electrons, the electrons may be captured by the sample molecules to create negatively charged ions. The voltage across the cell electrodes is pulsed to collect the remaining free electrons while the heavier ions are relatively unaffected and swept out the vent with the carrier gas flow.

Cell current is measured and compared to a reference current. The pulse rate is adjusted to maintain a constant cell current. The more uncaptured electrons, the lower the pulse frequency required to match the reference current. When a component that captures electrons passes through the cell, the pulse rate rises. This pulse rate is converted to a voltage and recorded.

The ^{63}Ni isotope

The radioactive isotope used in the cell is ^{63}Ni. It is plated onto the inner surface of the cell body and is solid at temperatures used in chromatography. Some other properties are listed in Table 9.

Table 9. Properties of ^{63}Ni

Half–life:	101.1 years
Emission:	65.87 keV max., beta radiation
Melting point:	1453°C
Dimensions of the active part of the ECD:	Inside diameter: 1.2 cm Height: 1 cm
Total activity ("regular" ECD):	555 MBq (15 millicuries) maximum

ECD licenses

Customers in the United states can purchase an ECD under either a General License or a Specific License. Customers outside the United States should contact their local Agilent sales office for information.

Specific License

Specific license ECDs require you to obtain a Materials License from the Nuclear Regulatory Commission (NRC) or the local state agency, permitting you to possess the amount and kind of radioisotope used in the detector. You can typically ship, sell, or transfer the ECD to other Specific Licensees. If the license permits, you may also open the ECD for cleaning.

General License

General License ECDs do not require a Materials License. You become a General Licensee automatically when you purchase an ECD directly from Agilent. Some states may require that you register the ECD with a state agency.

Certain restrictions apply to General Licenses:

1. Owners may not open the ECD cell.
2. Owners shall not modify the cell in any manner.
3. Owners shall not use any solvent, including water, to internally clean the cell.
4. Owners shall not interfere with or attempt to defeat the overheat circuitry that may be supplied with the ECD.
5. Owners shall not transfer the ECD to another person or another location except as described in the applicable Regulations.
6. Owners must perform a radioactive leak test at least every 6 months.
7. Owners must maintain records as required by your local Agency (the NRC or, in certain states, a state agency).
8. Owners must notify the Agency in case of incidents or failures that might lead to a hazardous condition.

Refer to publication "Information for General Licensees," part no. 5961-5664, for important information on regulatory requirements.

ECD warnings

Although beta particles at this energy level have little penetrating power —the surface layer of the skin or a few sheets of paper will stop most of them—they may be hazardous if the isotope is ingested or inhaled. For this reason the cell must be handled with care: Radioactive leak tests must be performed at the required intervals, the inlet and outlet fittings must be capped when the detector is not in use, corrosive chemicals must not be introduced into the detector, and the effluent from the detector must be vented outside the laboratory environment.

WARNING Materials that may react with the ^{63}Ni source, either to form volatile products or to cause physical degradation of the plated film, must be avoided. These materials include oxidizing compounds, acids, wet halogens, wet nitric acid, ammonium hydroxide, hydrogen sulfide, PCBs, and carbon monoxide. This list is not exhaustive but indicates the kinds of compounds that may cause damage to ^{63}Ni detectors.

WARNING In the *extremely* unlikely event that *both* the oven *and* the detector heated zone should go into thermal runaway (maximum, uncontrolled heating in excess of 400°C) at the *same* time, *and* that the detector remains exposed to this condition for *more than 12 hours*, take the following steps:

- After turning off the main power and allowing the instrument to cool, cap the detector inlet and exhaust vent openings. Wear disposable plastic gloves and observe normal laboratory safety precautions.
- Return the cell for exchange, following directions included with the License Verification Form (part no. 19233-90750).
- Include a letter stating the condition of abuse.

It is unlikely, even in this very unusual situation, that radioactive material will escape the cell. However, permanent damage to the ^{63}Ni plating within the cell is possible, and therefore, the cell must be returned for exchange.

WARNING Do not use solvents to clean the ECD.

WARNING You may not open the ECD cell unless authorized to do so by your local nuclear regulatory agency. Do not disturb the four socket-head bolts. These hold the cell halves together. Removing or disturbing them is a violation of the terms of the General License and could create a safety hazard.

Safety precautions when handling ECDs

- Never eat, drink, or smoke when handling ECDs.
- Always wear safety glasses when working with or near open ECDs.
- Wear protective clothing such as laboratory jackets, safety glasses, and gloves, and follow good laboratory practices. Wash hands thoroughly with a mild non-abrasive cleaner after handling ECDs.
- Cap the inlet and outlet fittings when the ECD is not in use.
- Connect the ECD exhaust vent to a fume hood or vent it to the outside. See the latest revision of title 10, Code of Federal Regulations, part 20, (including appendix B) or the applicable State regulation. For other countries, consult with the appropriate agency for equivalent requirements.

  Agilent Technologies recommends a vent line inside diameter of 6 mm (1/4-inch) or greater. With a line of this diameter, the length is not critical.

Part 2. General Information

Detector pneumatics

The ECD is available with or without electronic pneumatic control (EPC). Figure 21 and Figure 22 illustrate the pneumatics design for the electron capture detector with and without EPC.

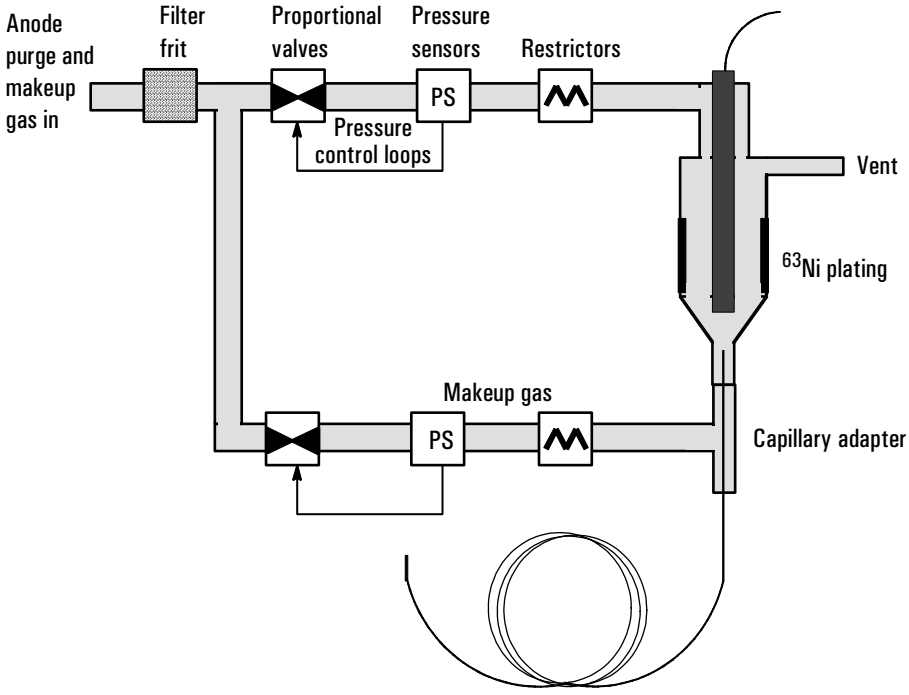


Figure 21. ECD pneumatics—EPC

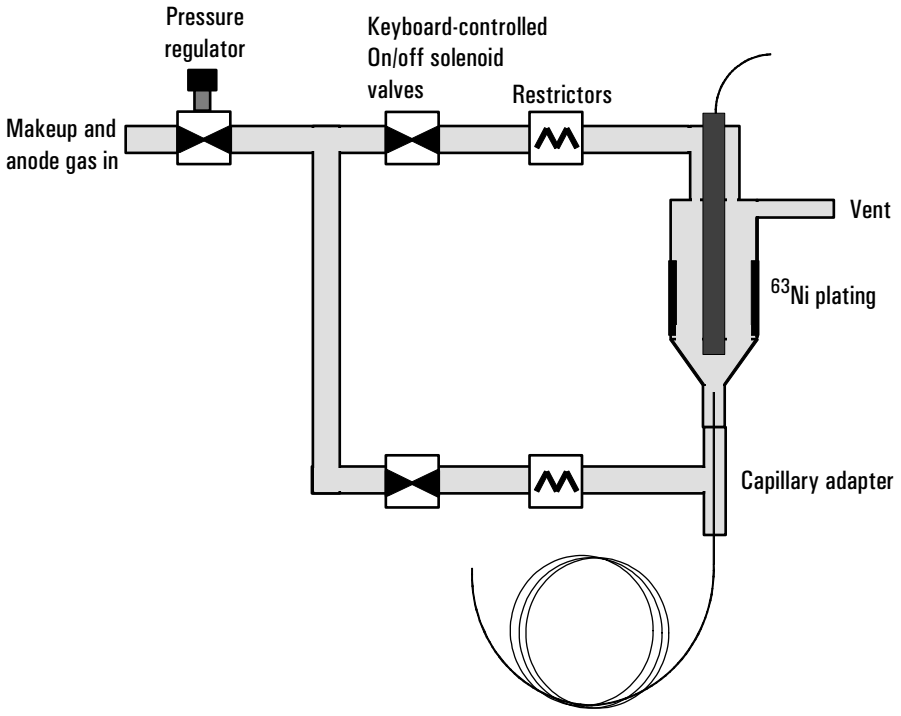


Figure 22. ECD pneumatics—NonEPC

Sensitivity

The response of the ECD depends upon many factors. These include the molecular composition of the analyte and its concentration, the cell cleanliness, the column, the inlet, and instrument setpoints (temperature and flow rates).

It is, therefore, important to create calibration curves on all compounds and evaluate detector response on a regular basis. Even with frequent calibration, you can expect the detector response to change when operating conditions change—for instance, when analyzing dirty samples that contaminate the detector.

Linearity

The ECD response factor versus concentration curve is nonlinear and the shape of the curve varies with the compound. A multipoint calibration is advised. When calibrating from the ChemStation, we suggest that you specify a *second-order curve fit.* When using an Agilent integrator, specify a *nonlinear fit.*

Gases

The ECD operates with either nitrogen or argon/methane as the makeup and anode gas.

Because of the high detector sensitivity, carrier and makeup gas must be dry and oxygen-free. High-quality moisture, chemical, and oxygen traps in good condition should be installed in carrier and makeup gas supply lines.

Detector electronics adjust according to the type of gas specified in the control tables. With the ECD cell, the anode purge gas should always be on. If you do turn it off, it is switched on when you initiate an `Adjust offset`.

Temperature

To prevent peak tailing and to keep the cell clean, the detector temperature should be set higher than the highest oven temperature used—the setpoint should be based on the elution temperature of the last compound. If you operate at excessively high temperatures, your results will not necessarily improve and you may increase sample and column decomposition.

Electrometer

The Configure Detector control table contains an `On/Off` setpoint for the `Electrometer`. Keep the electrometer on all the time when operating your detector.

Adjust offset

When you enter an `Adjust offset` setpoint, or press [On] to use the stored value, the reference current increases or decreases until the detector `Output` frequency is stable and equal to the `Adjust offset` value. Use this feature to achieve the same baseline on different ECDs or to ensure a consistent baseline from day to day. Before using `Adjust offset`:

Make a few runs.

Check that `Reference current` is greater than 0.5 nA and note the `Output`.

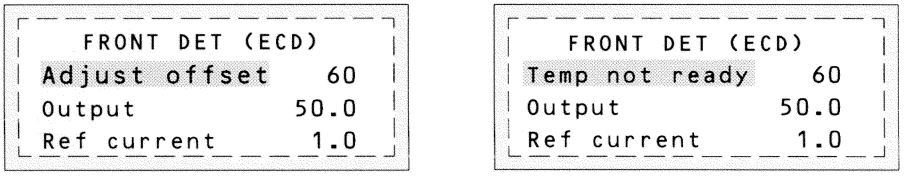
When the `Output`, which is typically very high for the first few runs, has stabilized, you can `Adjust offset` if desired.

The `Adjust offset` process takes about 30 seconds.

Example:

If the detector and oven temperature setpoints have not been reached, you see the following blinking display:

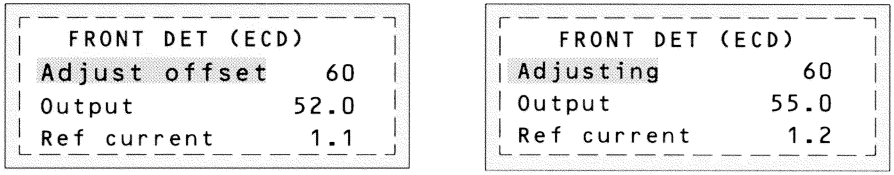
Detector on—detector or oven temperatures not at setpoint



= flashing display

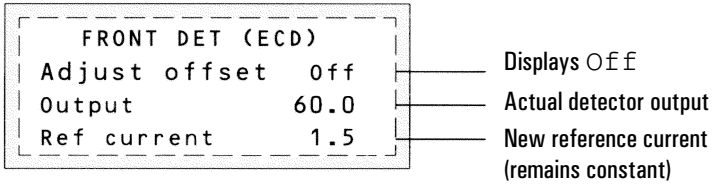
Once the temperature setpoints are reached, the `Adjust offset` process begins. If the `Anode gas` was off, it is switched on. You see the following blinking display as the `Ref current` changes:

Detector on—adjusting offset time



The detector is ready when the display stops blinking and the `Adjust offset` line is `Off`. The `Ref current` value remains constant until a new `Adjust offset` number is entered.

Detector ready



Aborting adjust offset

Press [Delete] or [Off] to abort the `Adjust offset` process.

Reference current

Once your detector is ready, check the reference current. It should be greater than 0.5 nA. If it is below 0.5 nA, enter a higher `Adjust offset` value.

You can enter a setpoint directly for the reference current, avoiding the series of steps involved in the `Adjust offset` process. Higher reference current setpoints are associated with higher `Output`.

Output—pulse interval

Each output unit on the ECD is equal to 5 Hz. The normal background frequency ranges from 20 to 120 on the display (100 to 600 Hz). If your Output exceeds 120 (600 Hz) while on baseline, you may want to check for a dirty cell, gas leaks, dirty makeup gas, traps, or tubing, and column bleed. See the detailed procedures for checking the output, baking out a dirty cell, and changing or conditioning traps later in this chapter.

If the `Output` while on baseline is less than 20 (100 Hz), sensitivity will be reduced. Use `Adjust offset` to increase the `Output`.

Part 3. Operating the ECD

Use the information in Figure 10 when selecting temperatures and flows. Choose a minimum source pressure from Figure 23. If you have an EPC detector, you must add 10 psi (69 kPa) to the source pressure on the chart.

Table 10. Operating Parameters

Gas type	Recommended flow range	Suggested flow
Carrier gas		
Packed columns *(nitrogen or argon-methane)*	30 to 60 mL/min	
Capillary columns *(hydrogen, nitrogen, or argon-methane)*	0.1 to 20 mL/min, depending on diameter	
Capillary makeup (nitrogen or argon-methane)	20 to 150 mL/min (50 to 60 mL/min typical. Adjust up or down to shift calibration curves.)	60 mL/min
Anode gas (same type as makeup gas)		
EPC detector		
Capillary columns	10% of makeup gas	6 mL/min
Packed columns	3 to 6 mL/min	
NonEPC detector		
Capillary columns	Not controlled by user. Flows are nominally 6 to 7% of makeup gas flow.	
Packed columns	3 to 6 mL/min	
Temperature		
250°C to 400°C		
Detector temperature is typically set 25°C greater than the highest oven ramp temperature.		
Adjust Offset		
30 to 70, range is 20 to 200		
Reference current		
≥0.5 nA, range is 0.5 nA to 5.0 nA		

Gas pressures

NonEPC detector—choose a flow, find a starting source pressure.
EPC detector—choose a flow, find a pressure. Set source pressure 10 psi higher.

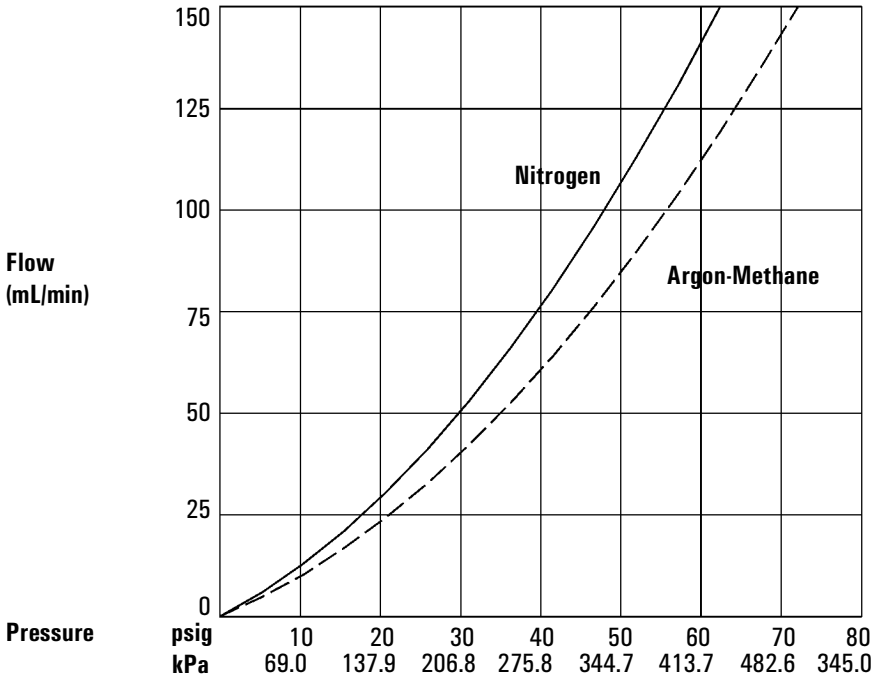
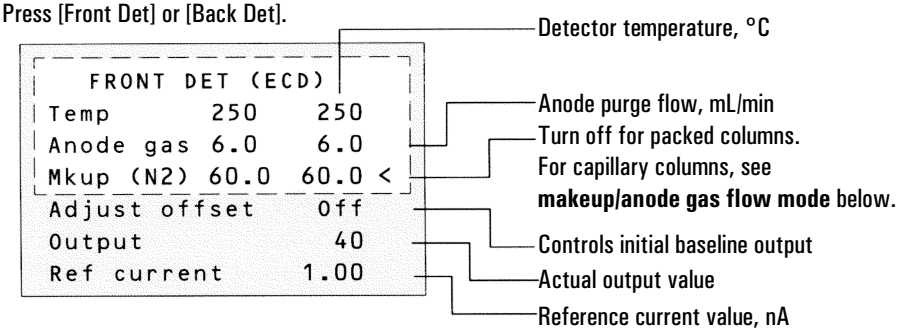


Figure 23. Pressure/flow relationships for ECD detector and makeup gases *(at 25°C and 1 atmosphere of pressure)*

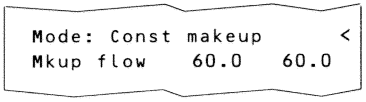
Operating with EPC

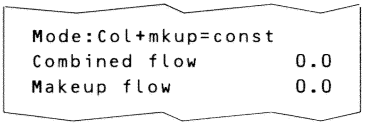
Press [Front Det] or [Back Det].



Makeup gas flow mode:

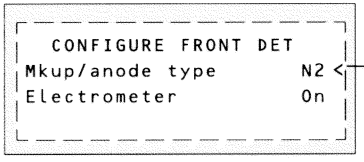
If configured for capillary columns, your control table will also include one of these:





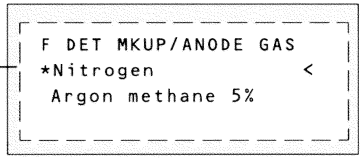
To **change makeup mode,** scroll to Mode: and press [Mode/Type].
Make a selection and enter the appropriate flow values.

To change **makeup/anode gas type,**
press [Config][Front Det] or
[Config][Back Det]:



Do not turn electrometer on or off.

Press [Mode/Type] to **change makeup gas**:



Select a gas and press [Enter].

Figure 24. ECD control table with EPC

Procedure: Using the ECD with EPC

Verify that your detector gases are connected, a column is properly installed, and the system is free of leaks. Set the oven temperature and the inlet temperature and flow. Make sure your carrier gas type ([Config][Inlet]) is the same as that plumbed to your GC.

1. Press [Front Det] or [Back Det] to open the ECD control table.
2. Set the detector temperature.

Caution Detector electronics depend on the correct gas configuration.

Short-cut procedure:
(assumes correct setpoints are stored)

1. Open detector control table.
2. Turn temperature On.
3. Turn makeup gas On, if needed.
4. Press [Det Control] and check Output.

3. Verify that the makeup/anode gas type is the same as that plumbed to your instrument. The gas type is in parentheses next to the Mkup line on the control table. Change the gas type, if necessary (page 7).
4. Enter a value for the anode gas.
5. Enter a value for the makeup gas.

   If you are using ***packed columns***, turn off the makeup gas.

   If your ***capillary column*** is *defined* and connected to an EPC inlet, choose a new flow mode (page 7), if desired, and set the makeup or combined gas flow.

   If your capillary column is *not defined* or connected to a nonEPC inlet, only constant flow is available. Enter a makeup gas flow.

Operating without EPC

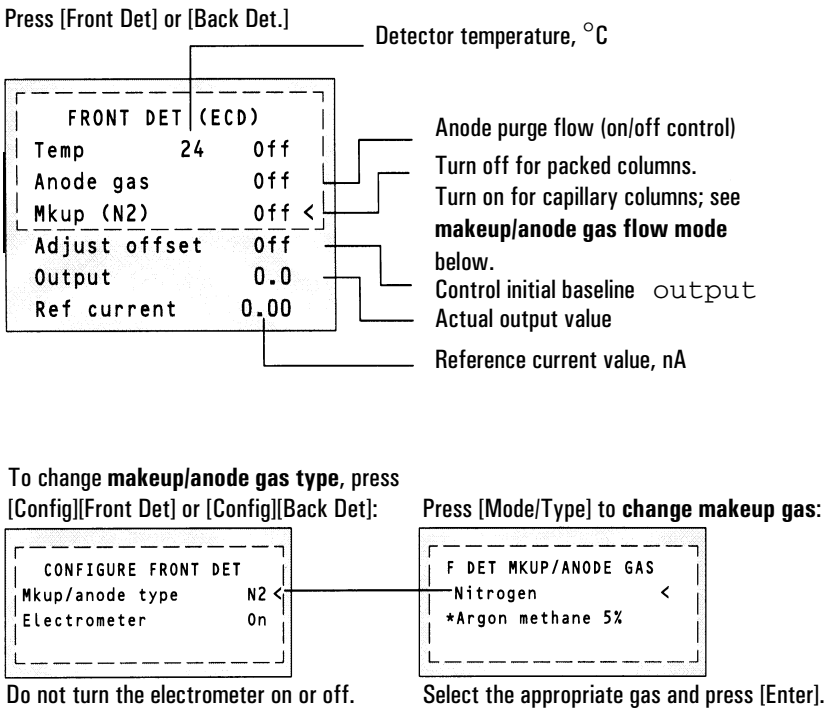


Figure 25. ECD control table without EPC

Procedure: Using the ECD without EPC

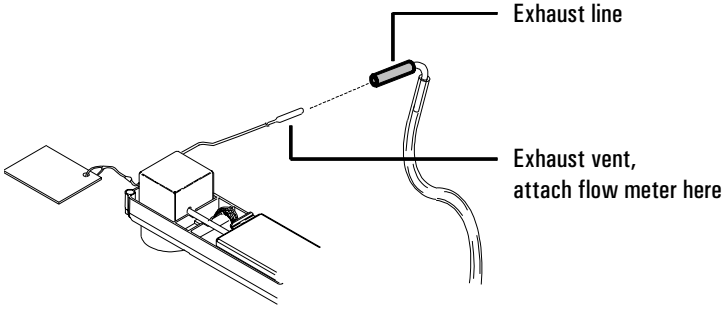
Before operating the ECD, be sure your detector gases are connected, a column is installed, and the system is free of leaks. Set oven temperature and inlet temperature and flow. Make sure your carrier gas type ([Config][Inlet]) is the same as that plumbed to your GC.

Use a flow meter with an ECD adapter to measure flows.

1. Press [Front Det] or [Back Det] to access the ECD control table.
2. Set the detector temperature.
3. Verify that the makeup/anode gas type is the same as that plumbed to your instrument. The gas type is in parentheses next to the Mkup line on the control table. Change the gas type, if necessary (page 7).

Caution Detector electronics depend on the correct gas configuration.

4. Disconnect the detector exhaust line from the detector exhaust vent so that you can attach a flow meter.

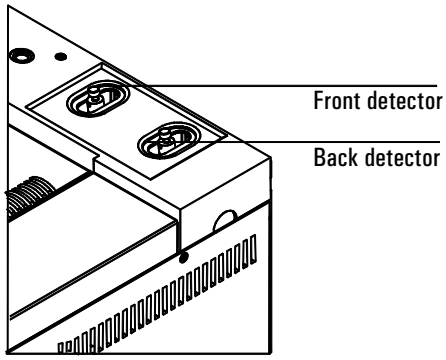


5. If you are using ***packed columns***, turn off the makeup gas from the control table, and set the anode gas flow:
   a. Turn `Anode gas` on from the control table.
   b. Turn on the supply pressure.
   c. Measure the flow out of the vent tube.

d. Locate the makeup/anode gas pressure regulator on top of the GC. *Clockwise* increases flow and *counterclockwise* decreases flow.

e. Adjust the pressure until the anode gas flow is correct. If you have flow through the column, subtract that from your total.

Makeup/anode gas pressure regulator



Short-cut procedure:
(assumes correct flows are set and setpoints are stored)

1. Open detector control table.
2. Turn temperature `On`.
3. Turn makeup gas `On`, if needed.
4. Press [Det Control] and check Output.

6. If you are using ***capillary columns,*** turn the makeup/anode gas on from the control table.

   a. Set the supply pressure.

   b. Measure the flow out of the vent tube.

   c. Locate the makeup/anode pressure regulator on top of the GC. *Clockwise* increases flow and *counterclockwise* decreases flow.

   d. Adjust the pressure with the until the makeup flow is correct. If you have flow through the column, subtract that from your total. The anode flow is 10% of the makeup flow.

7. Reconnect the detector exhaust line to the detector exhaust vent after making all flow measurements.

Part 4. Checkout Conditions and Chromatogram

This section contains a typical example of a test sample chromatogram. It may be used as a general guide to instrument performance.

Note that injection volumes listed with operating conditions do not necessarily indicate total absolute volume injected. Volume given is simply the graduation (plunger position) read from a standard 10 µL syringe. For a heated inlet, actual sample volume injected will also include an additional 0.4 to 0.7 µL, the volume of sample volatilized from inside the syringe needle. For the dedicated, on-column inlet (unheated), the syringe plunger position more accurately reflects the true injected volume.

Also note that the following procedure and results are intended only to provide evidence of a properly functioning inlet and/or detector system; they are not necessarily suitable to test a given system against its specification limits.

ECD checkout conditions

Column and sample

Type	5 30m × 0.32mm Q× 0.25 μm PN 19091J-413
Sample	ECD Checkout 18713-60040
Injection volume	1 μL

Inlet

Temperature	200°C Purged/Packed or Split/Splitless
	Oven Track Cool On-Column
	80°C PTV (see below)
Inlet pressure	25 psi (Constant pressure for EPC inlets, helium)

Split/Splitless

Mode	Splitless
Purge flow	60 mL/min
Purge time	0.75 min

Inlet, continued

PTV

Mode	Splitless
Inlet temperature	80°C
Initial time	0.1 min
Rate 1	720°C/min
Final temp 1	350°C
Final time 1	2 min
Rate 2	100°C/min
Final temp 2	250°C
Final time 2	0 min
Inlet pressure	25 psi (Constant pressure for EPC inlets)
Purge time	0.75 min
Purge flow	60 mL/min

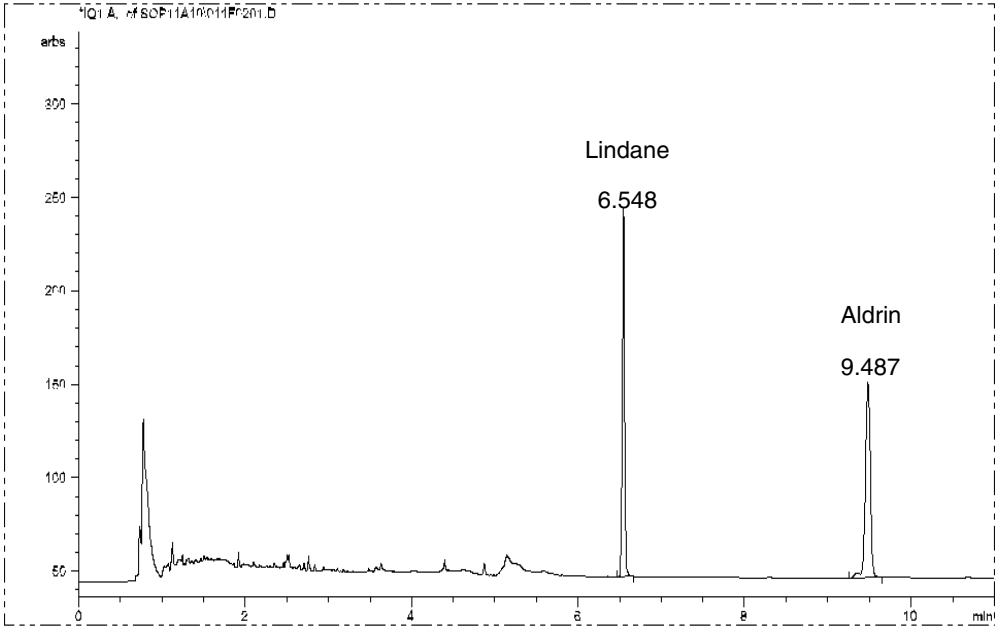
Detector

Temperature	300°C
Anode purge (N_2)	60 mL/min
Makeup	6 mL/min
Offset	Should be < 70 display counts

Oven

Initial temp	80°C
Initial time	0 min
Rate 1	15°C/min
Final temp	180°C
Final time	10 min

Typical ECD checkout chromatogram



Your retention times will differ but peaks should resemble the example.

Part 5. Maintaining the Detector

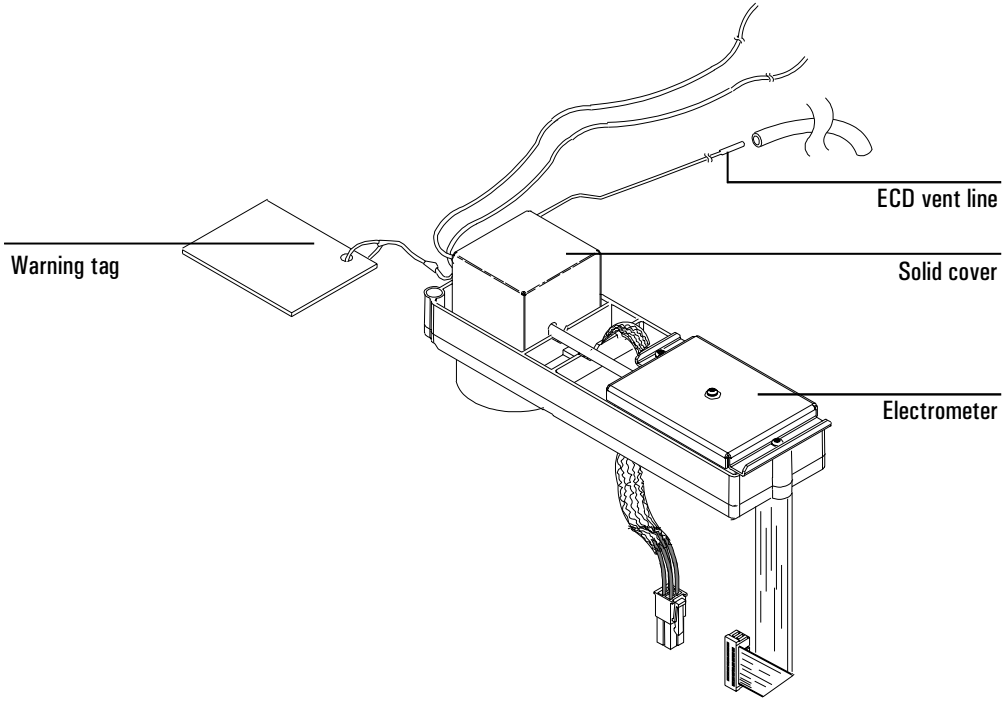


Figure 26. The ECD

Correcting performance problems

Performance problems, such as an output reading that is too high or too low or unsatisfactory chromatographic results (for example, a noisy baseline), can be caused by leaks or deposits in the detector or another part of the chromatographic system. To determine the location of the problem, you need to perform a series of tests.

Before testing the detector, however, consider the nature of the problem. If you have recently made a change to the GC system and are now seeing an elevated output level, there is a good chance that the change has either introduced contaminants or caused a leak in the system. For example, if you recently switched gas supplies, the new gas may contain impurities. Or if you recently installed a new column, there could be a leak at the detector fitting.

On the other hand, if the output value or noise level has been increasing gradually, the cause is likely to be a slow build-up of deposits. For example, the detector may contain contaminants from column bleed, or a trap may be saturated. If the change has been gradual, and if you have not modified the GC system recently, you can probably start by checking for contamination. *Note: Contamination in this procedure refers to non-radioactive deposits from such things as column bleed or dirty samples!*

1. Make sure the detector is operating under normal conditions and that at least 2 hours have lapsed since the last run.

   Check the output value in the detector control table. If it differs considerably from the normal output level—either it is higher or lower—you should continue with this procedure to identify the cause of the abnormal reading as a leak or contamination.

2. Use an electronic leak detector to check for leaks at the inlet and detector and the column fittings. Correct leaks, and then check the output level. If it is still abnormal, continue to step 3.

3. The detector itself is not a likely source of leaks, so you should leak test the inlet if the output reading is still abnormal. See the maintenance material for your inlet in the *Inlets* volume.

   If the inlet is not leaking, go to step 4 to check for leaks in the detector.

   If the inlet is leaking, correct the leaks and check the output. If it is still abnormal, the detector also may be leaking. Go to step 4.

4. Follow the leak test for the detector (page 138).

   If the detector is not leaking, the cause of the problem is contamination. Go to step 5.

   If the detector is leaking, correct the leaks, and then recheck the output. If it is still abnormal, go to step 5.

5. Check for contamination:

   a. Remove the column and plug the detector connection with the cap (part no. 19234-20650) and cap nut (part no. 19234-20570).

   b. Run the detector at your normal operating conditions but with only makeup gas only flowing through it. Monitor the output. If it is normal for your detector, then the contamination is from another part of the GC system. Go on to step 6.

   c. If the output is abnormal, then the detector is contaminated. Perform a thermal bake out to decontaminate the detector. Refer to the procedure on page 139.

6. One part at a time, check the rest of the GC system for contamination by making the following changes and monitoring the output:

   - Replace the column with an empty column and compare the output readings.
   - Switch to a different inlet (if possible), and compare the output.
   - Switch to a different source of gas and compare the output.
   - Replace the traps; compare the output.

Procedure: Checking for gas leaks

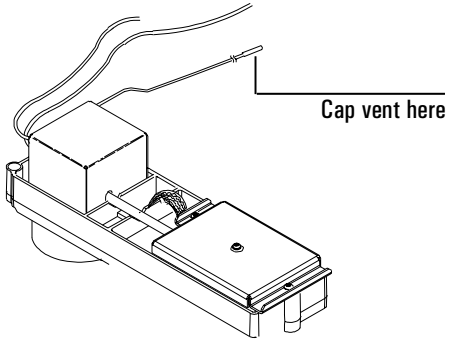
The detector is an unlikely leak source. If you suspect that there is a leak in your GC system, first leak check the gas plumbing to the GC, the inlet, and the column's inlet and detector connections. If all these areas pass their leak tests but you suspect that a leak is still present, follow this procedure to test the detector.

The oven and inlet should be at their normal operating temperature for this test.

Materials needed:

- A vent plug (part no. 5060-9055)
- An electronic leak detector capable of detecting your carrier gas

1. Turn off the inlet pressure. Allow some time to purge the system of the gas.
2. Turn off the anode and makeup gas flows.
3. Cap the ECD exhaust vent with the vent plug.



4. Set pressure at the inlet to 15 psi (103 kPa). Monitor the system pressure from the inlet. Allow time for the system to become fully pressurized (at least 1 minute). When the system is fully pressurized turn off the pressure or the gas. If you have a nonEPC inlet, turn off the pressure at the initial supply.

   Monitor the pressure for 10 to 15 minutes.

   If the pressure stays stable or drops only by 0.2 or 0.3 psi/min, you can consider the ECD leak-free. If pressure drops, you have a leak. Continue to step 5.

5. Use the electronic leak detector to check for leaks at the column fitting and plugged vent. If you find leaks, tighten the fittings, and repeat the leak test.

   If the other system components are leak-free, then the ECD may be leaking. The ECD cannot be disassembled without special license from the Nuclear Regulatory Commission or Agreement State Licensing Agency (USA only). Contact your Agilent service representative for more information.

Procedure: Thermal cleaning

If your baseline is noisy or the output value is abnormally high and you have determined that these problems are not being caused by leaks in the GC system, you may have contamination in the detector from column bleed. To remove contamination, you should perform a thermal cleaning (also called “bake-out”) of the detector.

WARNING Detector disassembly and/or cleaning procedures other than thermal should be performed only by personnel trained and licensed appropriately to handle radioactive materials. Trace amounts of radioactive ^{63}Ni may be removed during other procedures, causing possible hazardous exposure to β- and x-radiation.

WARNING To prevent possible hazardous contamination of the area with radioactive material, the detector exhaust vent always must be connected to a fume hood, or otherwise vented in compliance with the latest revision of Title 10, CFR, Part 20, or with state regulations with which the nuclear regulatory commission has entered into an agreement (USA only). For other countries, consult with the appropriate agency for equivalent requirements.

Materials needed:

- Cap for the detector connection (part no. 19234-20650)
- The nut to connect the cap (part no. 19234-20570)

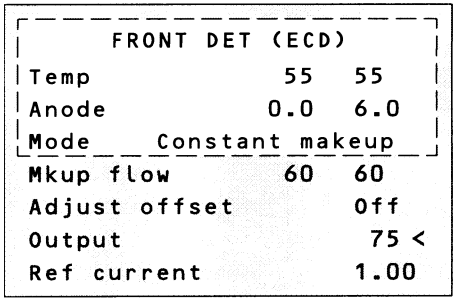
1. With the detector and oven at normal operating temperatures, press [Front Det] or [Back Det] to open the control table. Note the value of Output for later comparison.
2. Turn the anode purge and the makeup gas flow off.
3. Remove the column from the detector. Make sure to cap the unconnected end. Install a clean, unpacked glass or metal column.

4. Enter the following values:

Detector temperature	350 to 375°C
Anode purge	5 to 6 mL/min
Makeup gas	60 mL/min
Carrier gas	30 to 90 mL/min
Oven temperature	250°C

   Allow thermal cleaning to continue for several hours and then cool the system to normal operating temperatures.

5. Check the ECD output value on the control table. It should be lower than the first reading. If it is not contact your Agilent service representative.



Performing a wipe test (radioactivity leak test)

Electron capture detectors must be tested for radioactive leakage at least every 6 months. Records of tests and results must be maintained for possible inspection by the Nuclear Regulatory Commission and/or responsible state agency. More frequent tests may be conducted when necessary.

The procedure used is the **wipe test**. A wipe test kit is supplied with each new detector. Refer to the information card supplied in the Wipe Test Kit for instructions on performing the test.

6

The Micro-Cell Electron Capture Detector

Chapter 6
The Micro-Cell Electron Capture Detector

Part 1. Regulatory and Safety Information

Agilent Technologies provides two types of electron capture detectors. The “regular” detector, abbreviated as ECD, has a larger internal volume (approximately 10 times) than the micro-cell detector, abbreviated as µ-ECD. The two types can be distinguished by the top cover of the detector—the ECD has a solid cover and the µ-ECD has a perforated cover.

This chapter describes the micro-cell detector (µ-ECD). The "regular" detector (ECD) is described in the previous chapter.

The µ-ECD contains a cell plated with ^{63}Ni, a radioactive isotope. The ^{63}Ni releases β particles that collide with carrier gas molecules to produce low-energy electrons—each β particle produces approximately 100 electrons. The free electrons produce a small current—called the *reference* or *standing current*—that is collected and measured in a pulsed circuit.

When a sample component molecule comes into contact with the free electrons, the electrons may be captured by the sample molecules to create negatively charged ions. The voltage across the cell electrodes is pulsed to collect the remaining free electrons while the heavier ions are relatively unaffected and swept out the vent with the carrier gas flow.

Cell current is measured and compared to a reference current. The pulse rate is adjusted to maintain a constant cell current. The more uncaptured electrons, the lower the pulse frequency required to match the reference current. When a component that captures electrons passes through the cell, the pulse rate rises. This pulse rate is converted to a voltage and recorded.

The ^{63}Ni isotope

The radioactive isotope used in the cell is ^{63}Ni. It is plated onto the inner surface of the cell body and is solid at temperatures used in chromatography. Some other properties are listed in Table 11.

Table 11. Properties of ^{63}Ni

Half–life:	101.1 years
Emission:	65.87 keV max., beta radiation
Melting point:	1453°C
Dimensions of the active part of the μ-ECD:	Inside diameter: 6 mm Height: 4.2 mm
Total activity (μ-ECD cell):	555 MBq (15 millicuries) maximum

ECD licenses

Customers in the United states can purchase a µ-ECD under either a General License or a Specific License. Customers outside the United States should contact their local Agilent sales office for information.

Specific License

Specific license µ-ECDs require you to obtain a Materials License from the Nuclear Regulatory Commission (NRC) or the local state agency, permitting you to possess the amount and kind of radioisotope used in the detector. You can typically ship, sell, or transfer the µ-ECD to other Specific Licensees. If the license permits, you may also open the µ-ECD for cleaning.

General License

General License ECDs do not require a Materials License. You become a General Licensee automatically when you purchase a µ-ECD directly from Agilent Technologies . Some states may require that you register the µ-ECD with a state agency.

Certain restrictions apply to General Licenses:

1. Owners may not open the μ-ECD cell.
2. Owners shall not modify the cell in any manner.
3. Owners shall not use any solvent, including water, to internally clean the cell.
4. Owners shall not interfere with or attempt to defeat the overheat circuitry that may be supplied with the μ-ECD.
5. Owners shall not transfer the μ-ECD to another person or another location except as described in the applicable Regulations.
6. Owners must perform a radioactive leak test at least every 6 months.
7. Owners must maintain records as required by your local Agency (the NRC or, in certain states, a state agency).
8. Owners must notify the Agency in case of incidents or failures that might lead to a hazardous condition.

Additional information is available in the publication "Information for General Licensees," part no. 5961-5664.

μ-ECD warnings

Although beta particles at this energy level have little penetrating power —the surface layer of the skin or a few sheets of paper will stop most of them—they may be hazardous if the isotope is ingested or inhaled. For this reason the cell must be handled with care: Radioactive leak tests must be performed at the required intervals, the inlet and outlet fittings must be capped when the detector is not in use, corrosive chemicals must not be introduced into the detector, and the effluent from the detector must be vented outside the laboratory environment.

WARNING Materials that may react with the ^{63}Ni source, either to form volatile products or to cause physical degradation of the plated film, must be avoided. These materials include oxidizing compounds, acids, wet halogens, wet nitric acid, ammonium hydroxide, hydrogen sulfide, PCBs, and carbon monoxide. This list is not exhaustive but indicates the kinds of compounds that may cause damage to ^{63}Ni detectors.

WARNING In the *extremely* unlikely event that *both* the oven *and* the detector heated zone should go into thermal runaway (maximum, uncontrolled heating in excess of 400°C) at the *same* time, *and* that the detector remains exposed to this condition for *more than 12 hours*, take the following steps:

- After turning off the main power and allowing the instrument to cool, cap the detector inlet and exhaust vent openings. Wear disposable plastic gloves and observe normal laboratory safety precautions.
- Return the cell for exchange, following directions included with the License Verification Form (part no. 19233-90750).
- Include a letter stating the condition of abuse.

It is unlikely, even in this very unusual situation, that radioactive material will escape the cell. However, permanent damage to the ^{63}Ni plating within the cell is possible, and therefore, the cell must be returned for exchange.

WARNING Do not use solvents to clean the μ-ECD.

WARNING You may not open the μ-ECD cell unless authorized to do so by your local nuclear regulatory agency. Do not disturb the four socket-head bolts. These hold the cell halves together. Removing or disturbing them is a violation of the terms of the General License and could create a safety hazard.

Safety precautions when handling μ-ECDs

- Never eat, drink, or smoke when handling μ-ECDs.
- Always wear safety glasses when working with or near open μ-ECDs.
- Wear protective clothing such as laboratory jackets, safety glasses, and gloves, and follow good laboratory practices. Wash hands thoroughly with a mild non-abrasive cleaner after handling μ-ECDs.
- Cap the inlet and outlet fittings when the μ-ECD is not in use.
- Connect the μ-ECD exhaust vent to a fume hood or vent it to the outside. See the latest revision of title 10, Code of Federal Regulations, part 20, (including appendix B) or the applicable State regulation. For other countries, consult with the appropriate agency for equivalent requirements.

  Agilent Technologies recommends a vent line inside diameter of 6 mm (1/4-inch) or greater. With a line of this diameter, the length is not critical.

Part 2. General Information

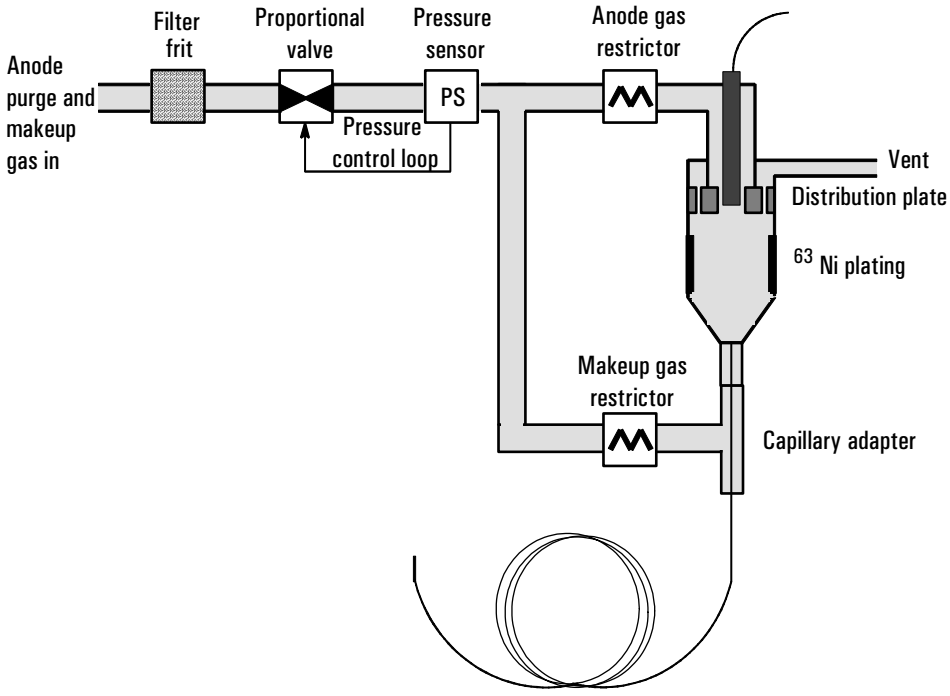


Figure 27. μ-ECD pneumatics—EPC only

Linearity

The μ-ECD response factor versus concentration curve is linear for four orders of magnitude or more (linear dynamic range = 10^4 or higher) for a broad range of compounds. You should still run a calibration curve on your samples to find the limits of the linear range for your materials.

Detector gas

The μ-ECD operates with either nitrogen or argon/methane as the makeup and anode gas.

Because of the high detector sensitivity, carrier and makeup gas must be dry and oxygen-free. Moisture, chemical, and oxygen traps in good condition should be installed in carrier and makeup gas supply lines.

Temperature

To prevent peak tailing and to keep the cell clean, the detector temperature should be set higher than the highest oven temperature used—the setpoint should be based on the elution temperature of the last compound. If you operate at excessively high temperatures, your results will not necessarily improve and you may increase sample and column decomposition.

Electrometer

The Configure Detector control table contains an `On/Off` setpoint for the `Electrometer`. Keep the electrometer on all the time when operating your detector.

Part 3. Operating the μ-ECD

If you intend to use the analog output from the μ-ECD, you must set the output Range to 10. This is done by pressing

[SIG 1] [RANGE] [10] [ENTER]

Use the information in Table 12 when selecting temperatures and flows. Maximum source pressure must not exceed 100 psi. Use the maximum source pressure to achieve maximum makeup flow rate.

Table 12. Operating Parameters

Gas	Recommended flow range
Carrier gas	
Packed columns *(nitrogen or argon-methane)*	30 to 60 mL/min
Capillary columns *(hydrogen, nitrogen, or argon-methane)*	0.1 to 20 mL/min, depending on diameter
Capillary makeup (nitrogen or argon-methane)	10 to 150 mL/min (30 to 60 mL/min typical.
Temperature	
250°C to 400°C	
Detector temperature is typically set 25°C greater than the highest oven ramp temperature.	

Notes

1. If the carrier gas type is different from the makeup gas type, the makeup gas flow rate must be at least three times the carrier gas flow rate.
2. μ-ECD sensitivity can be increased by reducing the makeup gas flow rate.
3. μ-ECD chromatographic speed (for fast peaks) can be increased by increasing the makeup gas flow rate.

Procedure: Operating the μ-ECD

Verify that your detector gases are connected, a column is properly installed, and the system is free of leaks. Set the oven temperature and the inlet temperature and flow. Make sure your carrier gas type ([Config][Inlet]) is the same as that plumbed to your GC.

1. Press [Front Det] or [Back Det] to open the µ-ECD control table.
2. Set the detector temperature. To keep the µ-ECD cell clean, this temperature must be higher than the oven temperature.

Caution Detector electronics depend on the correct gas configuration.

Short-cut procedure: *(assumes correct setpoints are stored)*

1. Open detector control table.
2. Turn temperature On.
3. Turn makeup gas On, if needed.
4. Press [Det Control] and check Output.

3. Verify that the makeup gas type is the same as that plumbed to your instrument. The gas type is in parentheses next to the `Mkup` line on the control table. Change the gas type, if necessary.
4. Enter a value for the makeup gas.

   If you are using ***packed columns***, turn off the makeup gas.

   If your ***capillary column*** is *defined* and connected to an EPC inlet, choose a new flow mode, if desired, and set the makeup or combined gas flow.

   If your capillary column is *not defined* or connected to a nonEPC inlet, only constant makeup flow is available. Enter a makeup gas flow.

Press [Front Det] or [Back Det]

```
FRONT DET (ECD)
Temp        250    250
Mkup (N2)  60.0   60.0 <
Output              40
```

Detector temperature, °C

Turn off for packed columns. For capillary columns, see **makeup gas flow mode** below.

Actual output value

Makeup gas flow mode:

If configured for capillary columns, your control table will also include one of these:

```
Mode: Const makeup       <
Mkup flow    60.0   60.0
```

```
Mode:Col+mkup=const
Combined flow          0.0
Makeup flow            0.0
```

To **change makeup mode,** scroll to Mode: and press [Mode/Type].

Make a selection and enter the appropriate flow values.

```
F DET MAKEUP MODE
*Const makeup flow
 Col+makeup=const    <
```

To change **makeup gas type**, press [Config][Front Det] or [Config][Back Det]:

```
CONFIGURE FRONT DET
Makeup gas type      N2 <
Electrometer         On
```

Do not turn the electrometer on or off.

Press [Mode/Type] to **change makeup gas**:

```
F DET MAKEUP GAS
*Nitrogen              <
 Argon methane 5%
```

Select a gas and press [Enter].

Figure 28. μ-ECD control table

Part 4. Checkout Conditions and Chromatogram

This section contains a typical example of a test sample chromatogram. It may be used as a general guide to instrument performance.

Note that injection volumes listed with operating conditions do not necessarily indicate total absolute volume injected. Volume given is simply the graduation (plunger position) read from a standard 10 µL syringe. For a heated inlet, actual sample volume injected will also include an additional 0.4-0.7 µL, the volume of sample volatilized from inside the syringe needle. For the dedicated, on-column inlet (unheated), the syringe plunger position more accurately reflects the true injected volume.

Also note that the following procedure and results are intended only to provide evidence of a properly functioning inlet and/or detector system; they are not necessarily suitable to test a given system against its specification limits

µ-ECD checkout conditions

Column and sample

Type	5 30m × 0.32mm × 0.25 μm PN 19091J-413
Sample	ECD Checkout 18713-60040
Injection volume	1 μL

Inlet

Temperature	200°C Purged packed
	250°C Split/splitless
	Oven Track Cool On-Column
	80°C PTV (see below)
Inlet pressure	25 psi (Constant pressure for EPC inlets, helium)

Split/Splitless

Mode	Splitless
Purge flow	60 mL/min
Purge time	0.75 min

Inlet, continued

PTV

Mode	Splitless
Inlet temperature	80°C
Initial time	0.1 min
Rate 1	720°C/min
Final temp 1	350°C
Final time 1	2 min
Rate 2	100°C/min
Final temp 2	250°C
Final time 2	0 min
Inlet pressure	25 psi (Constant pressure for EPC inlets)
Purge time	0.75 min
Purge flow	60 mL/min

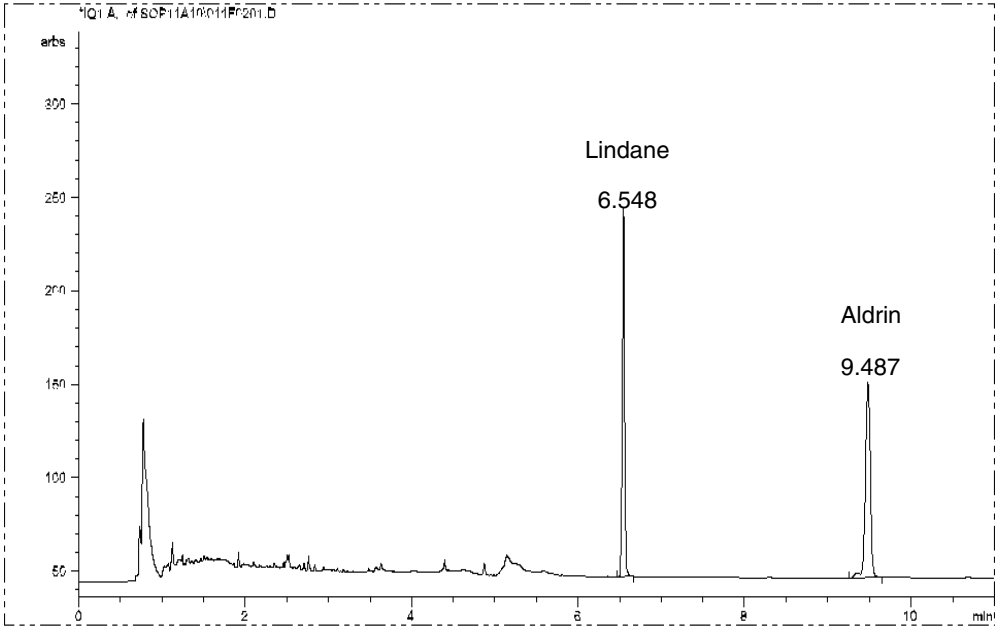
Detector

Temperature	300°C
Anode purge, nitrogen	60 mL/min
Makeup, nitrogen	25 ± 2 mL/min
Offset	Should be < 1000 display counts

Oven

Initial temp	80°C
Initial time	0 min
Rate 1	15°C/min
Final temp	180°C
Final time	10 min

Typical µ-ECD checkout chromatogram



Your retention times will differ but peaks should resemble the example.

Part 5. Maintaining the Detector

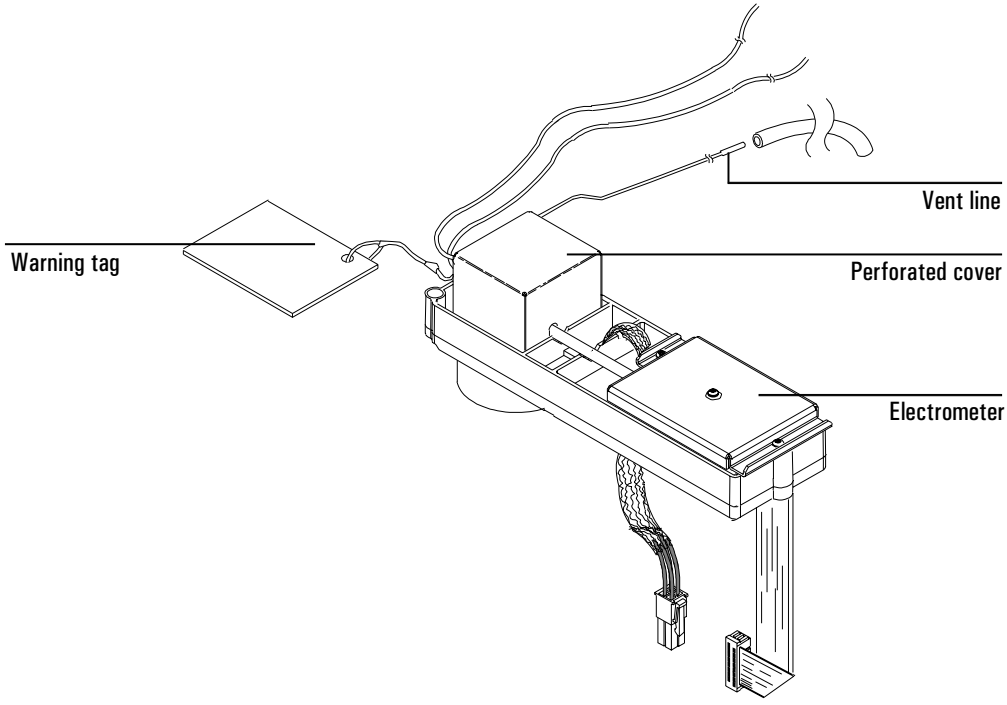


Figure 29. The μ-ECD

Correcting performance problems

Performance problems, such as an output reading that is too high or too low or unsatisfactory chromatographic results (for example, a noisy baseline), can be caused by leaks or deposits in the detector or other part of the chromatographic system. To determine the location of the problem, you need to perform a series of tests.

Before testing the detector, consider the nature of the problem. If you have recently made a change to the GC system and now see an elevated output level, there is a good chance that the change has either introduced contaminants or caused a leak in the system. For example, if you recently switched gas supplies, the new gas may contain impurities. Or if you recently installed a new column, there could be a leak at the detector fitting.

If the output value or noise level has been increasing gradually, the cause is probably a slow build-up of deposits. The detector may contain contaminants from column bleed or a trap may be saturated. If the change has been gradual and if you have not modified the GC system recently, you can probably start by checking for contamination. *Note: Contamination in this procedure refers to non-radioactive deposits from such things as column bleed or dirty samples!*

1. Make sure the detector is operating under normal conditions and that at least 2 hours have lapsed since the last run.

   Check the output value in the detector control table. If it differs considerably from the normal output level—either too high or too low—you should continue with this procedure to identify the cause of the abnormal reading.

2. Use an electronic leak detector to check for leaks at the inlet and detector and the column fittings. Correct leaks and then check the output level. If it is still abnormal, continue to step 3.

3. The detector itself is not a likely source of leaks, so you should leak test the inlet if the output reading is still abnormal. See the maintenance material for your inlet in the *Inlets* volume.

   If the inlet is not leaking, go to step 4 to check for leaks in the detector.

   If the inlet is leaking, correct the leaks and check the output. If it is still abnormal, the detector also may be leaking. Go to step 4.

4. Follow the leak test for the detector later in this document.

   If the detector is not leaking, the cause of the problem is contamination. Go to step 5.

   If the detector is leaking, correct the leaks, and then recheck the output. If it is still abnormal, go to step 5.

5. Check for contamination:

   a. Remove the column and plug the detector connection with the cap (part no. 19234-20650) and cap nut (part no. 19234-20570).

   b. Run the detector at your normal operating conditions but with only makeup gas flowing through it. Monitor the output. If it is normal for your detector, then the contamination is from another part of the GC system. Go on to step 6.

   c. If the output is abnormal, then the detector is contaminated. Perform a thermal bake out to decontaminate the detector. The procedure is later in this document.

6. One part at a time, check the rest of the GC system for contamination by making the following changes and monitoring the output:

   - Replace the column with an empty column and compare the output readings.
   - Switch to a different inlet (if possible), and compare the output.
   - Switch to a different source of gas and compare the output.
   - Replace the traps; compare the output.

Checking for gas leaks

The detector is an unlikely leak source. If you suspect that there is a leak in your GC system and have checked the gas plumbing to the GC, the inlet, and the column inlet and detector connections without finding it, follow this procedure to test the detector.

The oven and inlet should be at their normal operating temperatures.

Materials needed:

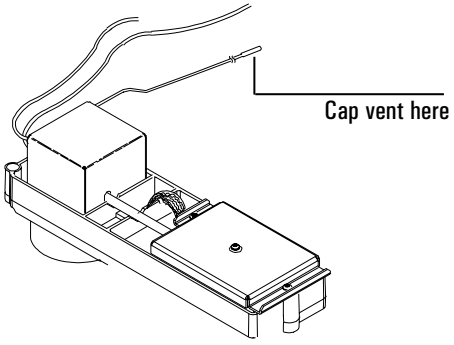
- A vent plug (part no. 5060-9055)
- An electronic leak detector capable of detecting your carrier gas

1. Turn off the inlet pressure. Allow some time to purge the system of the gas.
2. Turn off the makeup gas flow.

```
     FRONT DET (u-ECD)
Temp              150   150 <
Mkup flow         0.0   Off
Output            840000.0
```

When there is no flow, the output will be at its maximum, which is approximately 840,000 for both argon/methane and nitrogen

3. Cap the detector exhaust vent with the vent plug.



4. Set pressure at the inlet to 15 psi (103 kPa). Monitor the system pressure from the inlet. Allow time for the system to become fully pressurized (at least 1 minute). When the system is fully pressurized turn off the pressure or the gas. If you have a nonEPC inlet, turn off the pressure at the initial supply.

   Monitor the pressure for 10 to 15 minutes. If the pressure stays stable or drops only by 0.2 or 0.3 psi/min, you can consider the detector leak-free. If pressure drops, you have a leak. Continue to step 5.

5. Use the electronic leak detector to check for leaks at the column fitting and plugged vent. If you find leaks, tighten the fittings, and repeat the leak test.

   If the other system components are leak-free, then the detector may be leaking. The detector cannot be disassembled without special license from the Nuclear Regulatory Commission or Agreement State Licensing Agency (USA only). Contact your Agilent service representative for more information.

Thermal cleaning

If your baseline is noisy or the output value is abnormally high and you have determined that these problems are not being caused by leaks in the GC system, you may have contamination in the detector from column bleed. To remove contamination, you should perform a thermal cleaning (also called “bake-out”) of the detector.

WARNING Detector disassembly and/or cleaning procedures other than thermal should be performed only by personnel trained and licensed appropriately to handle radioactive materials. Trace amounts of radioactive ^{63}Ni may be removed during other procedures, causing possible hazardous exposure to β- and x-radiation.

WARNING To prevent possible hazardous contamination of the area with radioactive material, the detector exhaust vent always must be connected to a fume hood, or otherwise vented in compliance with the latest revision of Title 10, CFR, Part 20, or with state regulations with which the nuclear regulatory commission has entered into an agreement (USA only). For other countries, consult with the appropriate agency for equivalent requirements.

Materials needed:

- Cap for the detector connection (part no. 19234-20650)
- The nut to connect the cap (part no. 19234-20570)

1. With the detector and oven at normal operating temperatures, press [Front Det] or [Back Det] to open the control table. Note the value of Output for later comparison.
2. Turn the anode purge and the makeup gas flow off.
3. Remove the column from the detector. Make sure to cap the unconnected end. Install the detector cap and nut into the column detector fitting to plug the connection.

4. Enter the following values:
   - temperature = 350 to 375°C
   - makeup gas = 60 mL/min.
5. Set the oven temperature to 250°C.
6. Allow thermal cleaning to continue for several hours and then cool the system to normal operating temperatures.
7. Check the µ-ECD output value on the control table. It should be lower than the first reading. If it is not, contact your Agilent service representative.

Performing a wipe test (radioactivity leak test)

Electron capture detectors must be tested for radioactive leakage at least every 6 months. Records of tests and results must be maintained for possible inspection by the Nuclear Regulatory Commission and/or responsible state agency. More frequent tests may be conducted when necessary.

The procedure used is the **wipe test**. A wipe test kit is supplied with each new detector. Refer to the information card supplied in the Wipe Test Kit for instructions on performing the test.

7

The Flame Photometric Detector (FPD)

Chapter 7
The Flame Photometric Detector (FPD)

Part 1. General Information

The sample burns in a hydrogen-rich flame, where some species are reduced and excited. The gas flow moves the excited species to a cooler emission zone above the flame where they decay and emit light. A narrow bandpass filter selects light unique to one species, while a shield prevents intense carbon emission from reaching the photomultiplier tube (PMT).

The light strikes a photosensitive surface in the PMT where a light photon knocks loose an electron. The electron is amplified inside the PMT for an overall gain of up to a million.

The current from the PMT is amplified and digitized by the FPD electronics board. The signal is available either as a digital signal on the communications output or as a voltage signal on the analog output.

The FPD should not be stored at temperatures above 50°C, based on the original manufacturer's specifications for the PMT.

Linearity

Several mechanisms produce sulfur emission. The excited species is diatomic, so that emission intensity is approximately proportional to the square of the sulfur atom concentration.

The excited species in the phosphorus mode is monatomic, leading to a linear relationship between emission intensity and atom concentration.

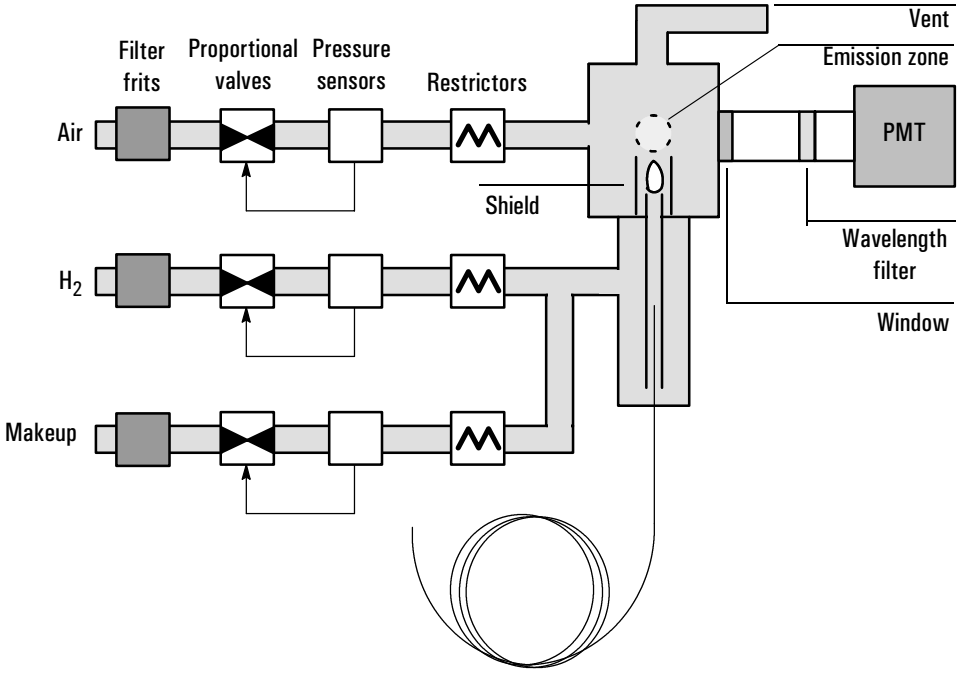


Figure 30. Schematic of a flame photometric detector

Quenching effects

Hydrocarbon quenching occurs when a high concentration of carbon dioxide from a hydrocarbon peak is in the flame at the same time as the sulfur species. Part of the light emitted by the sulfur species is absorbed by some CO_2 species.

Self-quenching occurs at high concentrations of the heteroatom species. Some other ground state (unactivated) species reabsorbs the emitted photon, preventing it from reaching the PMT.

These effects are reduced by good chromatographic practices. The column should provide good separation of the compounds, those that contain sulfur or phosphorus as well as those that do not but may absorb light. A careful, multilevel calibration is well worth the investment!

Detector and gas cleanliness must be maintained to have consistent responses. Since most sulfur and phosphorus compounds contain chemically active sites, the injection and column systems must be kept very clean.

PMT saturation

The photomultiplier tube may saturate if light intensity is too high. When this happens, increasing concentration produces little or no increase in signal and peak tops are rounded or flattened. Dilute the sample to correct the problem.

Optical filters

The filters are marked on the edge with the transmission wavelength. Each filter has a mirrored side—which must face the flame when installed—and a colored surface.

The sulfur filter is blue/purple and transmits at 393 nanometers.

The phosphorus filter is yellow/green and transmits at 525 nanometers.

Fused silica liner

The 6890 FPD uses an inert fused silica insert liner in the transfer line. This allows fused silica columns up to 530 µm ID to run right to the base of the flame, minimizing sample tailing or loss on chemically active sites. The liner is also compatible with standard packed columns.

Conditions that prevent the detector from operating

- Temperature set below 120°C
- Air or hydrogen flow set at `Off` or set at `0.0`
- Ignition failure

Detector shutdown

If a critical detector gas is shut down due to a pneumatics or ignition failure, your detector shuts down. This turns off everything except the detector temperature and makeup gas flow.

Compatibility requirements

If a single wavelength FPD is to be used with an Agilent ChemStation, the ChemStation must be version 4.02 or higher.

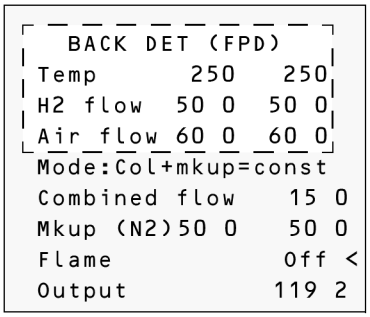
If a dual wavelength FPD is to be used with an Agilent ChemStation, the ChemStation must be version 5.01 or higher.

The dual wavelength FPD

This is a single burner module with two PMT housings, one with a sulfur filter and the other with a phosphorus filter. Because the optimum gas flows for these elements are quite different, performance of this detector is a compromise.

The detector mounts in the back position and is heated by the Back Det and AUX 2 heaters. The AUX 2 setpoint is automatically set by the Back Det setpoint.

Two signal channels and two electrometer boards are used, one for each PMT. The Back Det control table runs the detector, while the Front Det operates in a special "signal only" mode. Typical tables for a dual wavelength FPD are:



If a heated zone is assigned to the Front Det position, an "F det type mismatch" will be declared. To override this, press [Config], scroll to the `Instrument` line and press [Enter]. Scroll to the `F det` line, press [Mode/Type], and select `Sig only FPD`.

Part 2. Using the Detector

Detector temperature considerations

The FPD flame produces considerable water vapor. The detector must be operated above 120°C to prevent condensation.

Unnecessarily high temperatures can cause thermal decomposition of many thermally labile phosphorus and sulfur compounds.

Detector temperature can have a significant effect on sulfur sensitivity. If analyzing compounds with high boiling points, the detector temperature should be set to 25°C above the final oven temperature—if allowed by the temperature limit of 250°C.

Heater configuration

The FPD burner module has two heated zones, one for the detector body and one for the transfer line.

A single wavelength FPD can be mounted in either the front or back position. In the front position, it uses the Front Det and AUX 1 heaters. In the back position, it uses the Back Det and AUX 2 heaters. A second detector—possibly another FPD—can be mounted in the unused position.

A dual wavelength FPD—simultaneous detection of sulfur and phosphorus—must be mounted in the back position, where it uses the Back Det and AUX 2 heaters. A second detector cannot be mounted.

The software automatically sets the AUX heater to the same setpoint as the Det heater. You do not have to contend with two separate entries.

Lit offset

Lit offset is the expected difference between the FPD output with the flame lit and the output with the flame off. It is used to determine whether an attempted ignition has succeeded and to detect a flame-out condition.

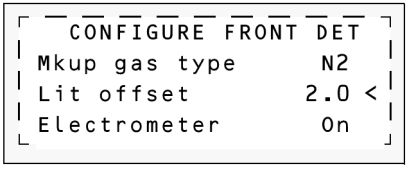
If the output with the flame on minus the output with the flame off is greater than `Lit offset`, the flame is considered lit.

The default setting for `Lit offset` is 2.0 picoamps. This is a good working value for all but very clean gases and systems. You may want to change this setpoint if:

- Your detector is attempting to reignite when the flame is still on, thus producing a shutdown.
- Your detector is not trying to reignite when the flame is out.

Procedure: Changing the Lit offset setpoint

1. Press [Config][Front Det] or [Config][Back Det].



2. Scroll to `Lit offset` and enter a number.
   The default is 2.0 pA.
   Enter 0 to disable the automatic reignite function.
   The setpoint range is 0 to 99.9 pA.

Flame ignition sequence

When either of the flame ignition methods on the next page is used, the 6890 FPD automatically performs this sequence:

1. Turns all detector gases—air, hydrogen, makeup—off. Carrier remains on.
2. Sets air flow to 200 mL/min.
3. Turns the glow plug ignitor on.
4. Ramps the hydrogen flow from 10 to 70 mL/min.
5. Resets the air flow to the air flow setpoint.
6. Resets the hydrogen flow to the hydrogen flow setpoint.
7. Turns the makeup gas on.
8. Compares the signal change with the `Lit offset` value. If the change is greater than `Lit offset`, declares the flame on (lit). If it is less, declares the flame off (not lit).

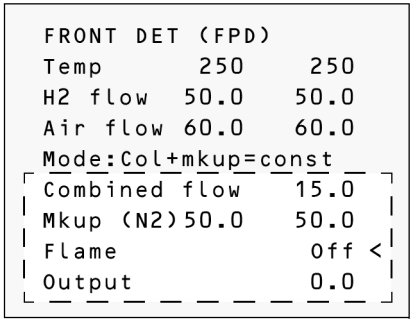
For this process to work, there must be enough air pressure to the pneumatics module to provide 200 mL/min flow. We recommend a supply pressure of 90 psi.

Lighting the flame

Manual

To start the flame ignition sequence:

Press [Front Det] or [Back Det]



Scroll to Flame and press [On]

Automatic

If the FPD output with the flame on falls below the flame-off output plus the `Lit offset` value, this is interpreted as a flame-out condition. The FPD runs the flame ignition sequence to relight the flame. If this fails, it runs the sequence again. If the second attempt also fails, the detector shuts down all functions except temperature and makeup gas flow.

Electrometer on/off

The Configure Detector control table contains an Electrometer On/Off setpoint.

On	High voltage and signal processing circuits are on. **If the photomultiplier tube is exposed to room light with the electrometer on, the tube will be destroyed.**
Off	High voltage and signal processing circuits are off. In this condition, it is safe to expose the photomultiplier tube to room light.

Caution Always turn the electrometer off before removing the PMT housing to avoid destroying the tube.

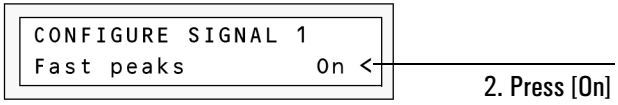
Electrometer data rates

Analog output for the FPD can be presented at either of two speeds. The faster speed allows minimum peak widths of 0.004 minutes, while the standard speed allows peak widths of 0.01 minutes.

Procedure: Using fast peaks

If you are using the *fast peaks* feature, your integrator must be fast enough to process the data coming from the GC. It is recommended that your integrator bandwidth be at least 15 Hz. To use fast peaks:

1. Press [Config][Signal 1] or [Config][Signal 2]



The fast peaks feature does not apply to digital output.

Operating the FPD

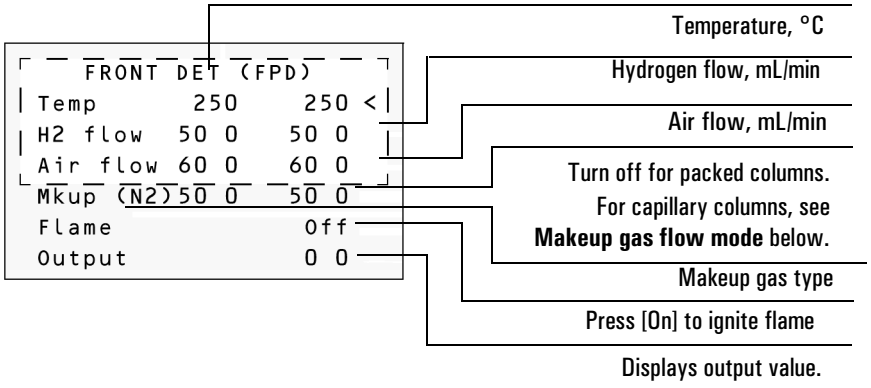
Table 13 gives the flows for the maximum sensitivity FPD flame, which is hydrogen-rich and oxygen-poor. It is difficult to light the flame with these flows, particularly in the sulfur mode. Helium, used as carrier or makeup gas, may cool the detector gases below the ignition temperature. We recommend using nitrogen rather than helium.

Table 13. Recommended Temperature and Flow

	Sulfur mode flows mL/min	Phosphorus mode flows mL/min
Carrier (hydrogen, helium, nitrogen, argon)		
Packed columns	10 to 60	10 to 60
Capillary columns	1 to 5	1 to 5
Detector gases		
Hydrogen	50	150
Air	60	110
Carrier + makeup	60	60
Supply pressure		
Air supply pressure: at least 90 psi for the ignition sequence. All others: adequate to achieve desired flows.		
Detector temperature		
Below 120°C, flame will not light.		
Set temperature about 25°C higher than highest oven temperature—limit is 250°C.		
Lit offset [Config] [Front Det] **or** [Back Det]		
If the detector output (with the flame on) minus the output (with the flame off) falls below this value, the FPD attempts to re-ignite twice. If output does not increase by at least this much, the detector shuts down.		
The recommended setting is 2.0 pA. A setting of 0 or [Off] disables autoignition.		

If the flame will not light with the sulfur mode flows shown, change to the phosphorus mode values. After the flame lights, gradually reduce the flows toward the sulfur mode values. Some experimentation will be required to find flows for your particular detector.

Press [Front Det] or [Back Det].



Makeup gas flow mode: If column dimensions are specified, the control table will also include one of these sets.

```
Mode:Const makeup   <          Mode:Col+mkup=const
Mkup flow 0.0     Off          Combined flow     0.0
                               Makeup flow       0.0
```

To **change makeup mode**, scroll to Mode: and press [Mode/Type].
Make a selection and enter the appropriate flow values.

```
 F DET MAKEUP MODE
*Const makeup flow
 Col+makeup=const  <
```

To view **makeup gas** or change **Lit offset**, press [Config][Front Det] or [Config][Back Det]:

```
 CONFIGURE FRONT DET
Mkup gas type      N2 <
Lit offset         2 0
Electrometer       On
```

It is not necessary to turn the electrometer on or off unless you are performing a maintenance procedure.

To change **makeup gas** type, press [Mode/Type]:

```
 FRONT DET MAKEUP GAS
 Helium                <
*Nitrogen
```

Select the appropriate gas.

Figure 31. FPD control table

Procedure: Using the FPD

Verify that all detector gases are connected, a column is installed, and the system is free of leaks. Check the oven temperature, inlet temperature, and column flow.

WARNING Verify that a column is installed or the FPD column fitting is plugged before turning on the air or hydrogen. An explosion may occur if air and hydrogen are allowed to leak into the oven.

1. Press [Front Det] or [Back Det] to open the FPD control table.
2. Set the detector temperature. The temperature must be greater than 120°C for the flame to light.
3. Change the hydrogen flow rate, if desired, and press [Off].
4. Change the air flow rate, if desired, and press [Off].
5. If you are using **packed columns**, turn off the makeup gas and proceed to Step 7.
6. If you are using **capillary columns**:
   a. Verify that makeup gas type is the same as that plumbed to your instrument (next to `Mkup` line of control table). Change the gas type, if necessary.
   b. If your capillary column is *defined* and is connected to an EPC inlet, choose a new flow mode, if desired, and set the makeup gas flow or combined flow.
   c. If your capillary column is *not defined* or is connected to a nonEPC inlet, enter a makeup gas flow. Only constant flow is available.
7. Scroll to `Flame` and press [On]. This turns on the air and hydrogen and initiates the ignition sequence. On ignition, the signal increases. Typical levels are 4 to 40 pA in sulfur mode, 10 to 70 pA in phosphorus mode. Verify that the flame is lit by holding a cold, shiny surface, such as a mirror or chrome-plated wrench, over the vent exit. Steady condensation indicates that the flame is lit.

Part 3. Checkout Conditions and Chromatogram

This section contains typical examples of test sample chromatograms. It may be used as a general guide to instrument performance.

Note that injection volumes listed with operating conditions do not necessarily indicate total absolute volume injected. Volume given is simply the graduation (plunger position) read from a standard 10 µL syringe. For a heated inlet, actual sample volume injected will also include an additional 0.4 to 0.7 µL, the volume of sample volatilized from inside the syringe needle. For the dedicated, on-column inlet (unheated), the syringe plunger position more accurately reflects the true injected volume.

Also note that the following procedure and results are intended only to provide evidence of a properly functioning inlet and/or detector system; they are not necessarily suitable to test a given system against its specification limits.

FPD checkout conditions

Column and sample

Type	5 30m × 0.32mm × 0.25 μm PN 19091J-413
Sample	FPD Checkout 8500-3697
Injection volume	1 μL

Inlet

Temperature	250°C Purged/Packed or Split/Splitless
	Oven Track Cool On-Column
	80°C PTV (see below)
Inlet pressure	25 psi (Constant pressure for EPC inlets, helium)

Split/Splitless

Mode	Splitless
Purge flow	60 mL/min
Purge time	0.75 min

Inlet, continued

PTV

Mode	Splitless
Inlet temperature	80°C
Initial time	0.1 min
Rate 1	720°C/min
Final temp 1	350°C
Final time 1	2 min
Rate 2	100°C/min
Final temp 2	250°C
Final time 2	0 min
Inlet pressure	25 psi (constant pressure for EPV inlets)
Purge time	0.75 min
Purge flow	60 mL/min

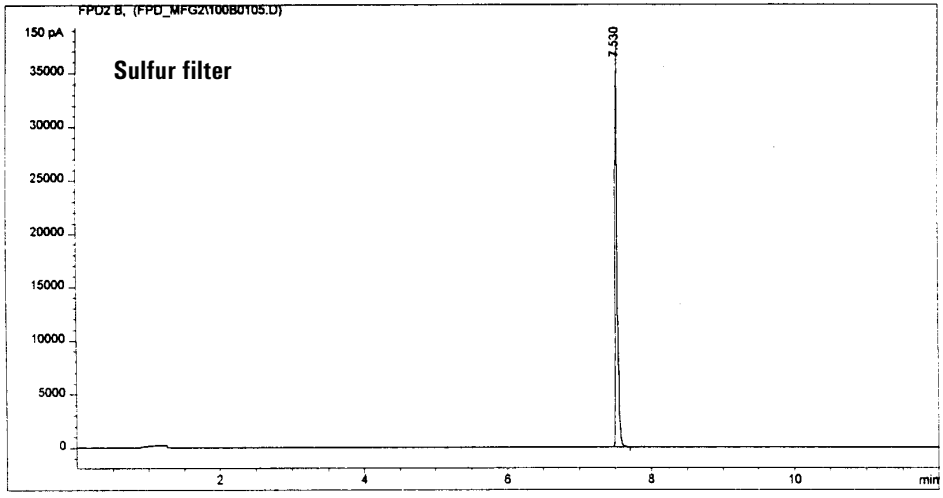
Detector

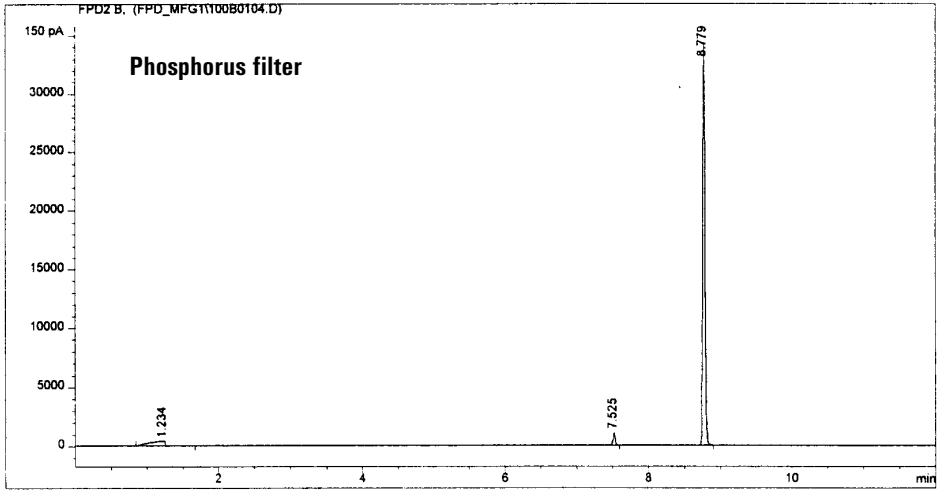
Temperature	250°C
Hydrogen flow	75 ± 2 mL/min
Air flow	100 ± 2 mL/min
Makeup flow	60 ± 2 mL/min, nitrogen
Offset, flow off (O-fa)	Should be < 40 display units
Offset, flame on (O + fb)	< [(O-fa) + 85 display units]

Oven

Initial temp	60°C
Initial time	0 min
Rate 1	25°C/min
Final temp	110°C
Final time 0 min	
Rate 2	10°C/min
Final temp 2	170°C
Final temp 1	3 min

Typical FPD checkout chromatograms





Your retention times will differ, but peaks should resemble this example.

Part 4. Maintaining the Detector

Caution Do not store the FPD at temperatures above 50°C, since this may damage the PMT.

Flame ignition problems

If the FPD flame won't light or stay lit, check/do the following:

1. Be sure there is a problem. Ignition is best confirmed by holding a mirror or shiny surface near the aluminum exhaust tube, with the rubber drip tube removed, and observing condensation if the flame is lit.
2. Check `Lit offset`. If it is zero, autoignition is turned off. If it is too large, the GC will not know that the flame is lit and will shut the detector down.
3. Increase the air supply pressure to the pneumatics module. This makes the flame easier to light but does not affect the air flow rate setpoint.
4. If the flame doesn't light at all, check the glow plug circuit. Observe the visual display, which will momentarily go to greater than 65500 counts when the flame lights. If the display doesn't change, check the pin connections at the printed circuit board, the lead connection on the glow plug and the appropriate 5A fuse on the GC main circuit board. If the glow plug has failed, replace it with part no. 0854-0141.
5. The flame is easier to light at higher detector temperatures.
6. Under some operating conditions, the flame may be more easily lit with the rubber drip tube removed. After lighting the flame, reinstall the drip tube.
7. If the flame still won't light, there could be a large leak in the system. This results in measured flow rates being different from actual flow rates, causing non-ideal ignition conditions. Thoroughly leak check the whole system.
8. If the analysis permits, substitute nitrogen for helium as carrier and makeup.
9. Increase hydrogen and air flow rates until ignition occurs, then reduce them toward the Table 13 values. Experiment for the best values.

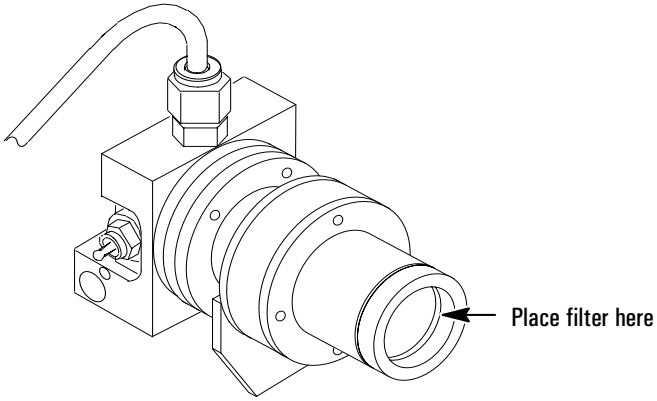
Changing wavelength filters

Install the correct optical filter, depending on the choice of Sulfur or Phosphorus mode. For Sulfur Mode, use the 393 nanometer filter (part no. 19256-80000). For Phosphorus Mode, use the 525 nanometer filter (part no. 19256-80010).

To change the filter:

WARNING Turn the main power switch—located under the left side of the oven door—off. If the photomultiplier tube is exposed to room light with the power on, it will be destroyed.

1. Release the retaining spring around the photomultiplier housing.
2. Pull the photomultiplier housing off the detector body. A twisting motion helps.
3. Remove the old filter. Use tissue to avoid fingerprints.
4. Place the new filter in the recess so that the silvered side faces the flame.



5. Push the PMT housing as far onto the detector body as it will go.
6. Install the retaining spring around the housing.
7. Restore power.

Leak testing

Turn off all supply gases. Cap the detector exhaust tube with a 1/4-inch Swagelok plug (part no. 0100-0196) and a 40% graphitized Vespel ferrule (part no. 0100-1061).

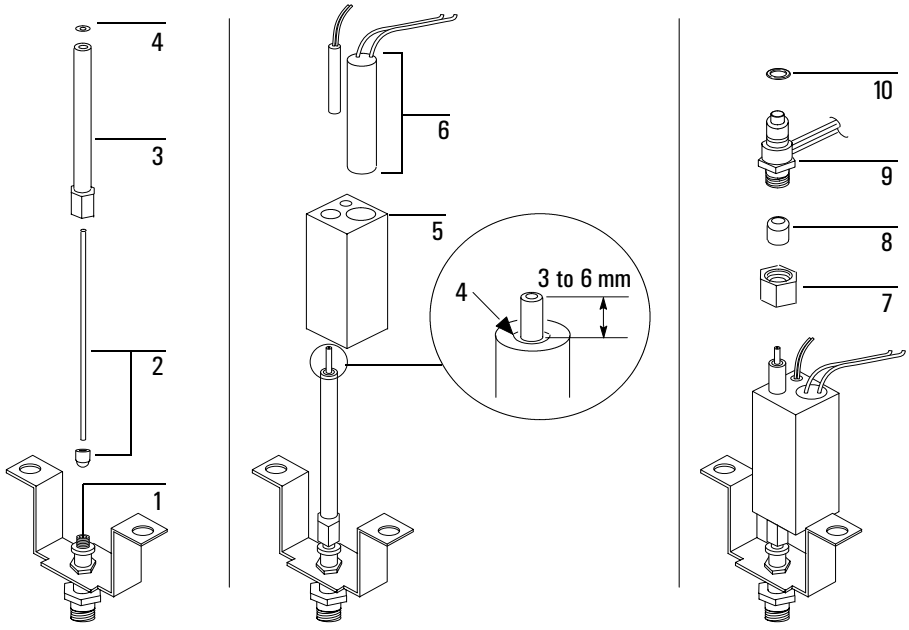
Caution When testing the flow system under pressure, do not exceed 210 kPa (30 psig). Higher pressures may damage the detector block window or seals.

Turn one of the gases on for a few seconds and then turn it off. The indicated flow-which is really a pressure-should remain constant or drop slowly. If not, there is a leak in the system. Begin checking possible leak sources and monitor the flow number to determine when the leak has been eliminated.

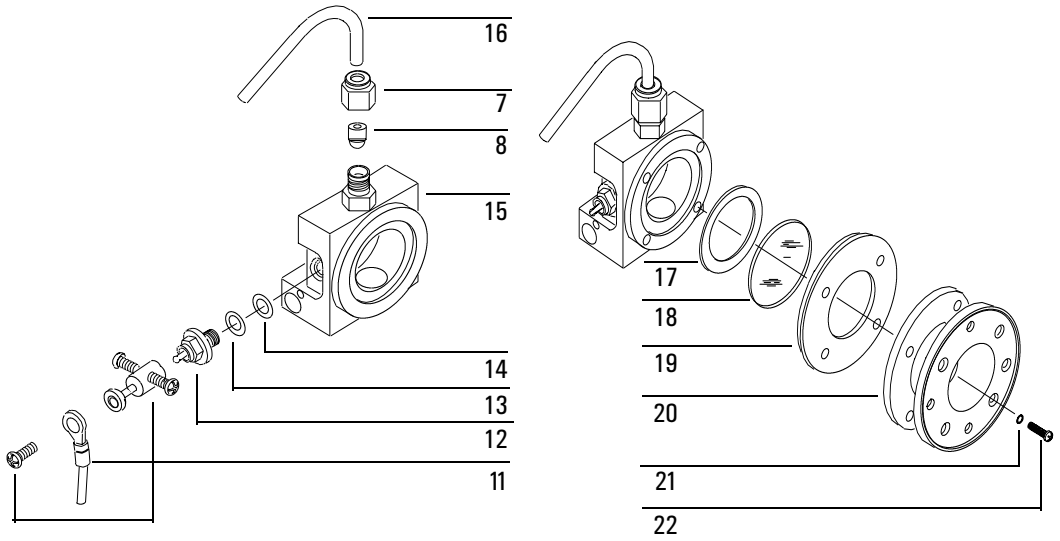
Possible leak sources, in order of decreasing probability, are:

1. Septum
2. Column fittings
3. Supply line swage-type plumbing connections
4. Detector block O-ring or Vespel seals
5. Other system plumbing

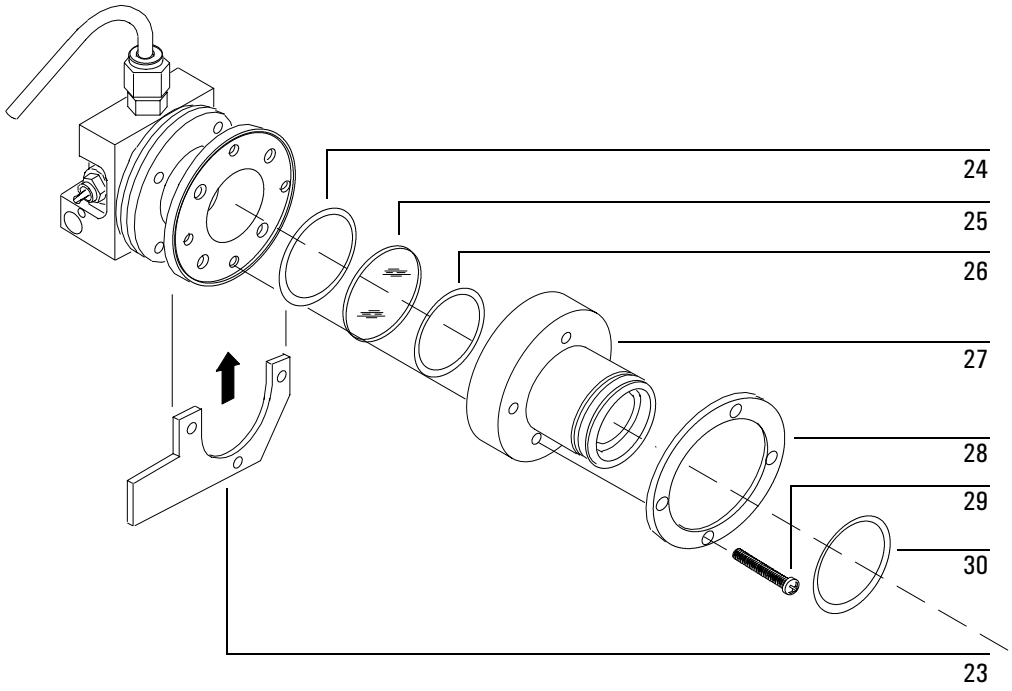
Parts identification



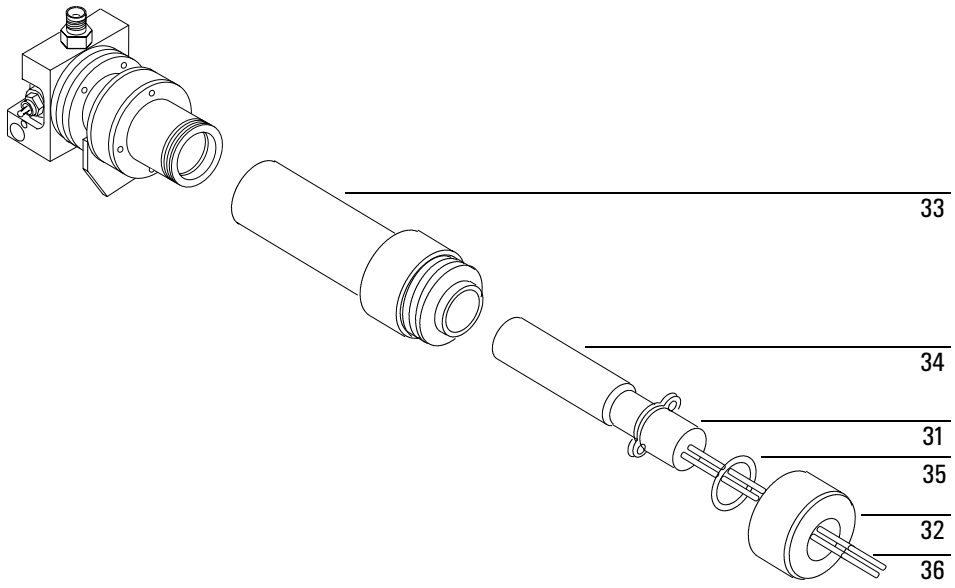
Item	Description	Part no.
1	Base assembly weldment	
2	Gigabore liner/ferrule assembly	19256-60590
3	Transfer tube	19256-80550
4	O-ring, Kalrez, transfer tube	0905-1101
5	Lower heater block	
6	Heater/sensor assembly	
7	Nut, brass, 1/4-inch	0100-0056
8	Ferrule, Vespel, 1/4-inch ID	5080-8774
9	Jet cartridge	G1535-80500
10	O-ring, Kalrez, jet cartridge	0905-1103



Item	Description	Part no.
7	Nut, brass, 1/4-inch	0100-0056
8	Ferrule, Vespel, 1/4-inch ID	5080-8774
11	Ignitor cable assembly	G1535-60600
12	Glow plug	0854-0141
13	Spacer, ignitor	19256-20590
14	O-ring, Kalrez, ignitor	0905-1102
15	Weldment, block	
16	Exit tube assembly, aluminum Exit tube assembly, stainless steel	19256-20700 19256-20705
17	Gasket, head shield	19256-80040
18	Window, first heat shield	19256-80030
19	Disk, heat shield	19256-20580
20	Coupling, stainless steel	19256-20550
21	Lockwasher (4 required)	2190-0108
22	Screw, M3 x 12 (4 required)	0515-0911



Item	Description	Part no.
23	Clamp	19256-00090
24	O-ring, silicone, 0.926-inch ID (orange)	0905-0955
25	Window, second heat shield	19256-80060
26	O-ring, silicone, 1.05-inch ID (orange)	0905-1104
27	Flange adapter	
28	Flange ring	19256-00200
29	Screw, M3 x 25 (4 required)	0515-0065
30	O-ring, Viton, 1.239-inch ID (brown)	0905-1100
Filters (not shown)		
	Sulfur mode	19256-80000
	Phosphorus mode	19256-80010



Item	Description	Part no.
31	Tube socket	19256-20670
32	End cap	19256-20710
33	PMT tube housing	19256-20650
34	Replacement photomultiplier tubes	
	PM tube ONLY	G1535-80050
	PM tube and housing assembly	19256-60510
35	O-ring for PM tube	0905-1099
36	Resistor network cable assembly	19256-60580

Cleaning/replacing windows, filters, and seals

Column bleed and/or effluent can contaminate the first quartz window (heat shield) nearest the detector module. Dust, fingerprints, atmospheric contaminants can dirty both quartz windows, the filter, and/or the photomultiplier tube window. Contamination anywhere along the light path between flame and PMT can reduce detector sensitivity.

1. Turn the electrometer off.
2. Turn hydrogen, air, and auxiliary gas supplies to the detector off. Turn the heaters off. Wait for the detector to cool.
3. Release the retaining spring around the photomultiplier housing.

Caution Always turn the electrometer off before removing the PMT housing to avoid destroying the tube.

Caution Keep the open end of the PMT housing covered as much as possible to avoid light damage to the tube.

4. Pull the PMT housing off the detector and remove the filter. Use lint-free lens tissue to clean the filter on both sides. Clean the PMT window seen inside the housing. Avoid scratching surfaces; do not use a cleaning fluid that might leave a film upon drying.
5. Inspect the filter: chips, scratches, and/or cracks in the light path scatter light, reducing detector sensitivity. Replace filters as necessary.

   Inspect the PMT window for damage; if necessary, replace the PMT.

   a. Remove the four screws in the PMT adapter flange and remove the flange. Use care as a quartz window is exposed and may fall out. Clean the window using lens tissue.

   b. Remove the four screws in the stainless steel coupling and carefully remove the coupling. The remaining quartz window may fall out. Clean the window using lens tissue.

Caution This window—the one closest to the flame—may stick when the detector is cold. It is easier to remove when the detector is warm, but be careful to avoid burns.

6. Note the placement and types of seals found on the disassembled parts. Seals should be replaced with new parts on reassembly.
7. Inspect the windows: chips, scratches, cracks or fogging in the light path scatter light, reducing sensitivity. Replace windows if necessary.
8. Reassemble the parts in reverse order, making sure all seals are of the proper type and in their proper locations. Tighten screws evenly and firmly to ensure gas- and light-tight seals. If the filter has a silvered side, it should face the flame (indicator arrows > on edge of filter should point toward the PMT).

Cleaning/replacing the jet

If a response problem is encountered (sensitivity, noise, selectivity) the FPD jet should be inspected for deposits and, if necessary, cleaned or replaced. To properly service the jet, the detector module should be removed from the instrument, followed by appropriate service:

1. Turn off power to the gas chromatograph and disconnect the main power cord. Remove the detector covers.
2. Allow time for the heated zones to cool to safe temperatures.

Caution Always turn the electrometer or the main power off before removing the PMT housing to avoid destroying the tube.

Caution Keep the open end of the PMT housing covered as much as possible to avoid light damage to the tube.

3. Remove the photomultiplier tube assembly from the detector module; also remove the filter. Set both in a safe place.
4. Remove the exhaust tubing.
5. Remove the sheet metal cover. On the single wavelength detector, it is held by two screws at the top and two at the bottom; on the dual wavelength detector it is held by two screws at the top. Loosen the screws holding the detector to the U-clamp. Use two wrenches to loosen the swage connection at the bottom of the jet assembly from the transfer line tube and carefully lift the burner module from the transfer tube so as not to damage the fused silica liner.

   It is not necessary to disconnect any plumbing, ignitor leads or the heater/sensor. Leave all attached and disconnect the detector block from the transfer line at the 1/4-inch swage fitting, then gently lift the block and rotate it enough to access the jet.

6. Remove and inspect the jet assembly. Rotating it slightly helps to free it. The jet assembly slips out of the FPD block more easily if the block is still warm. Use a wire or brush to remove any deposits.
7. This is also an ideal time to inspect/clean the glow plug (see "Flame ignition problems" on page 182), and inspect/clean the quartz windows (see "Cleaning/replacing windows, filters, and seals" on page 189).
8. Use compressed air or nitrogen to blow out loose particles from the jet and/or detector module body.
9. Inspect and clean deposits from the jet bore using a suitable wire. If the jet is damaged in any way, replace it. It is good practice to replace the jet, rather than try to clean it, particularly when extremely high sensitivity is required.
10. Install a new Kalrez O-ring seal onto the jet. Do not re-use the old O-ring.

Caution Be careful not to crush or side-load the fused silica liner when reinstalling the detector.

11. Reassemble all parts of the detector module; reassemble the module onto the instrument. Use a new Vespel ferrule to seal the detector module to the transfer line.
12. Reinstall the PMT assembly on the detector module; restore instrument gases and power.

Replacing the transfer line fused silica liner

Occasionally the transfer line fused silica liner between the column and FPD module must be inspected, cleaned, and/or replaced.

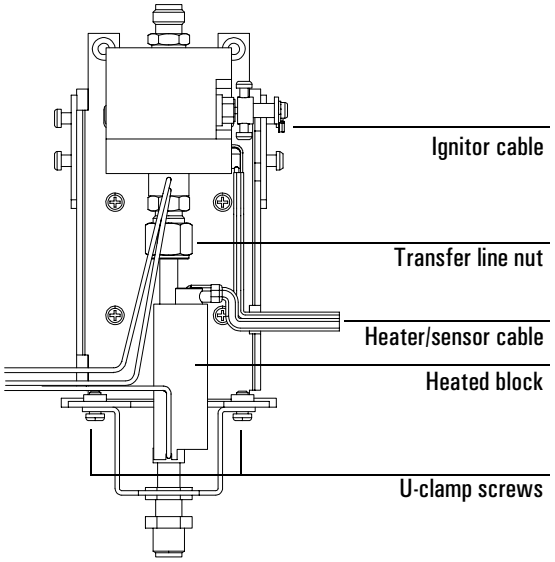
1. Turn off power to the gas chromatograph and disconnect the main power cord. Remove the detector covers.
2. Allow time for heated zones to cool to safe temperatures.
3. Inside the oven, remove the column to the FPD.

Caution Always turn the electrometer or the main power off before removing the PMT housing to avoid destroying the tube.

Caution In the next step, keep the open end of the PMT housing covered as much as possible to avoid light damage to the tube.

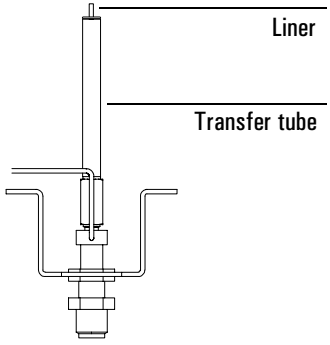
4. Remove the photomultiplier tube assembly—or assemblies—from the detector module; also remove the filter(s). Set them in a safe place.

5. Locate the ignitor cable attached to the side of the detector. Trace the cable back to the printed circuit board and disconnect it there.



6. Remove the exhaust tubing and the sheet metal cover—on the single wavelength detector, it is held by two screws on the top and two at the bottom; on the dual wavelength detector, it is held by two screws at the top.
7. Remove the four screws that attach the detector to the top of the oven (one at each corner). Remove the detector from the GC.
8. Loosen the transfer line nut. Remove the two screws that secure the U-clamp to the detector frame. Remove the U-clamp and the attached parts from the bottom of the detector.

9. Remove the transfer line nut and its ferrule, the heater/sensor cable assembly, and the heated block.



10. With an open end wrench, unscrew the transfer tube from the detector base. Lift the transfer tube—containing the fused silica liner—vertically off the instrument. Remove the fused silica liner and the 1/16-inch Vespel ferrule by pulling the liner and ferrule out from the bottom. Inspect for damage.
11. If necessary, install a new fused silica liner and Vespel ferrule. When doing so, carefully feed the fused silica liner through the Kalrez O-ring at the top of the transfer line so as not to damage the O-ring.
12. Carefully replace the fused silica liner, ferrule and tube onto the detector base. The fused silica liner should be positioned so that it protrudes 6 to 7 mm (1/4-inch) above the top of the transfer tube weldment. With a wrench, firmly tighten the transfer tube (1/2-turn past finger tight).
13. Reinstall the heated block, the heater/sensor cable assembly, the nut, and the ferrule. The notch in the bottom of the block fits over the tubing coming from the detector fitting.
14. Tighten the U-clamp screws, then tighten the nut on the transfer tube.
15. Place the detector on top of the instrument, orient it properly, and install the four screws to hold it. Install the top cover and the exhaust tubing.
16. Connect the ignitor cable to the printed circuit board.
17. Install the PMT assembly (or assemblies).
18. Restore normal operating conditions.

Replacing the photomultiplier tube

If the PMT is defective (high voltage on and the flame lit: low or no signal and/or high noise not attributed to any other source such as bad cables, light leaks, high temperature, defective signal board, etc.), it must be replaced.

1. Turn off power to the gas chromatograph and disconnect the main power cord.

Caution Turn the electrometer or main power off before opening the PMT housing to avoid destroying the tube.

2. Free the cables to the PMT from the clip on the support. Pull a few inches of the cables through the cable tie toward the end cap. Unscrew the end cap from the PMT assembly. Slide the cap away from the assembly.
3. Slide the resistor network cable assembly and the photomultiplier tube and socket out of the housing until about 1 inch of the tube is exposed.

Caution Protect the new PMT from light as much as possible to avoid damage to the tube.

4. Pull the socket off the PMT. Remove the PMT and replace with a new tube.
5. When seating the socket on the new tube, be certain that the missing pin on the tube base is lined up with the gap in the socket contacts.
6. Reassemble in reverse order. Make sure grease, fingerprints, dust, etc. are removed from the PMT window facing the detector module. Be sure that the O-ring is in place on the PMT/resistor bridge network assembly, as this is a critical light seal. If the O-ring is damaged, replace it.
7. Screw the end cap onto the PMT assembly. Pull the cables through the cable tie to eliminate slack at the end of the assembly. Place the cables in the clip on the side of the PMT housing support.

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