22 The Flame Ionization Detector

The Flame Ionization Detector

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 73 illustrates the pneumatics design for the FID.

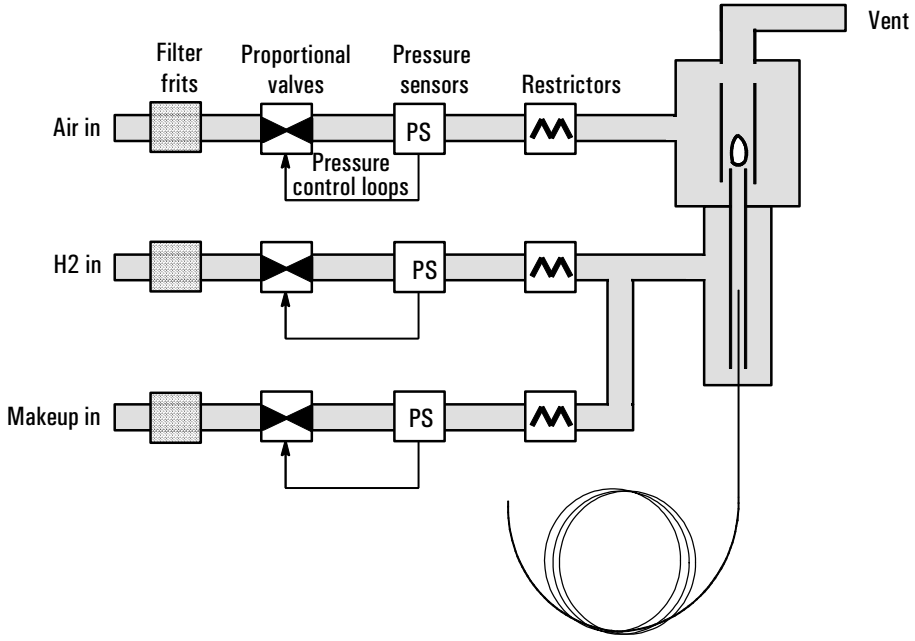


Figure 73 Schematic of a flame ionization detector

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.

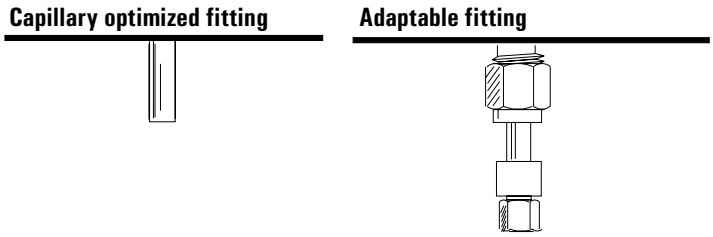


Table 59 Jets for the Capillary-Optimized FID

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 60 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 in "Replacing or cleaning the jet".

Automatic reignition—Lit offset

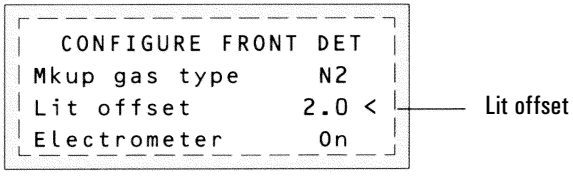
`Lit offset` is the expected difference between the FID output with the flame lit and the output with the flame off. If the output falls below this value, the FID will attempt to reignite twice. If the output does not increase by at least this value, the detector shuts down all functions except temperature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

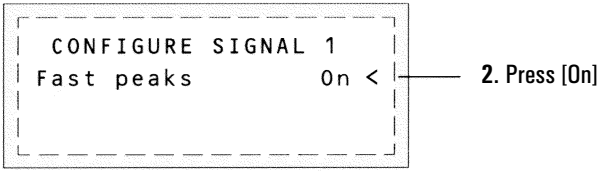
Data rates

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. Consult "Signal Handling".

The fast peaks feature does not apply to digital output.

Operating the FID

Use the information in Table 61 when selecting temperatures and flows. Choose a minimum source pressure from Figure 74.

Table 61 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

Choose a flow, find a pressure. Set source pressure 10 psi (70 kPa) higher.

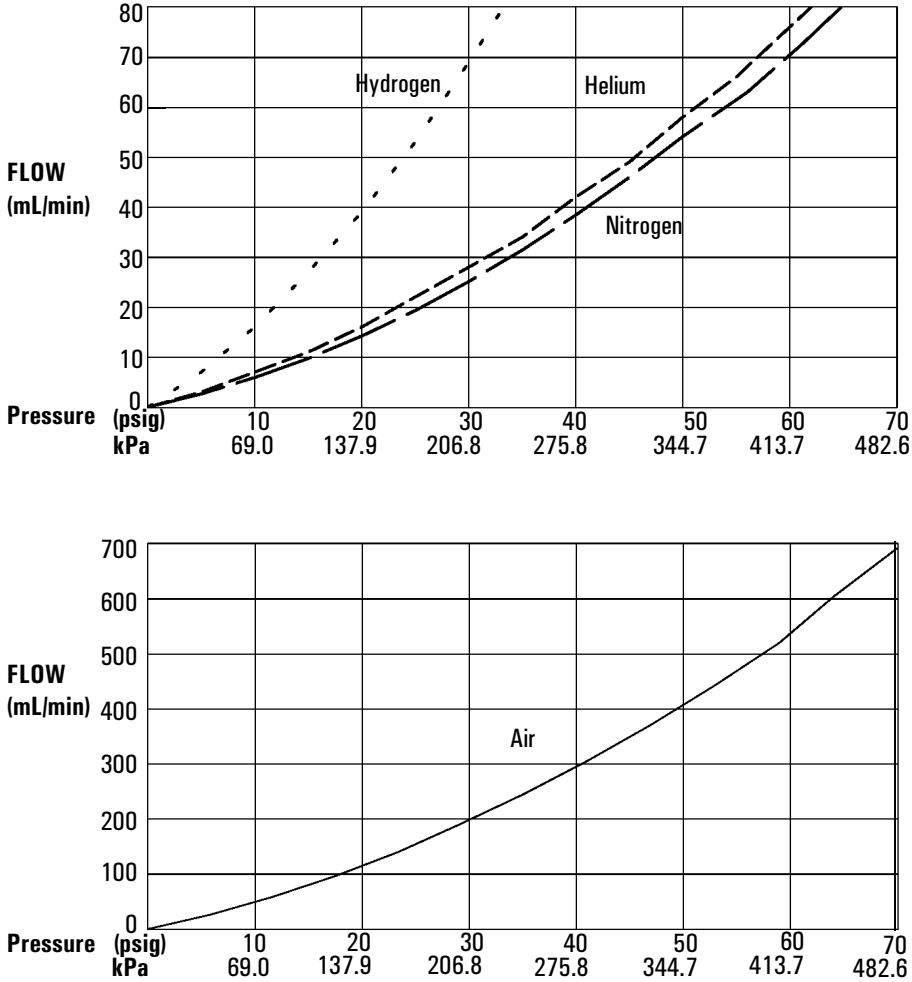
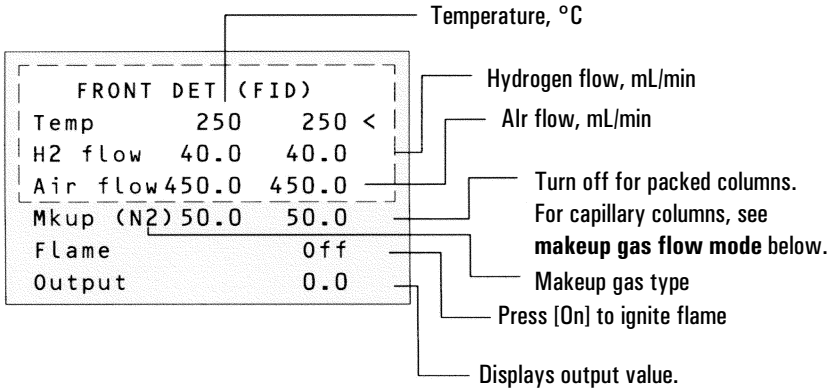


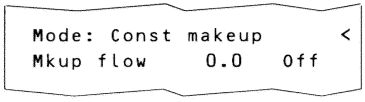
Figure 74 Typical pressure/flow relationships for FID gases
(at 25°C and 1 atmosphere of pressure)

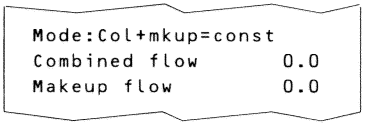
Operating with EPC

Press [Front Det] or [Back Det].

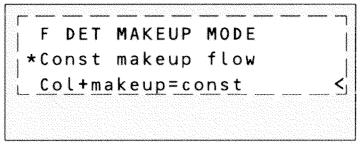


Makeup gas flow mode:
If column dimensions are specified, the control table will also include one of these:

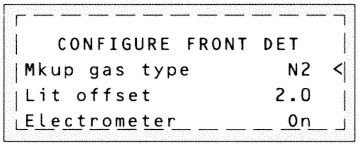




To **change makeup mode,** scroll to Mode: and press [Mode/Type]. Make a selection and enter the appropriate flow values.



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 75 FID control table

Procedure: Using the FID

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 75 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].

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	HP-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, 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)
Purge time	0.75 min
Purge flow	60 mL/min

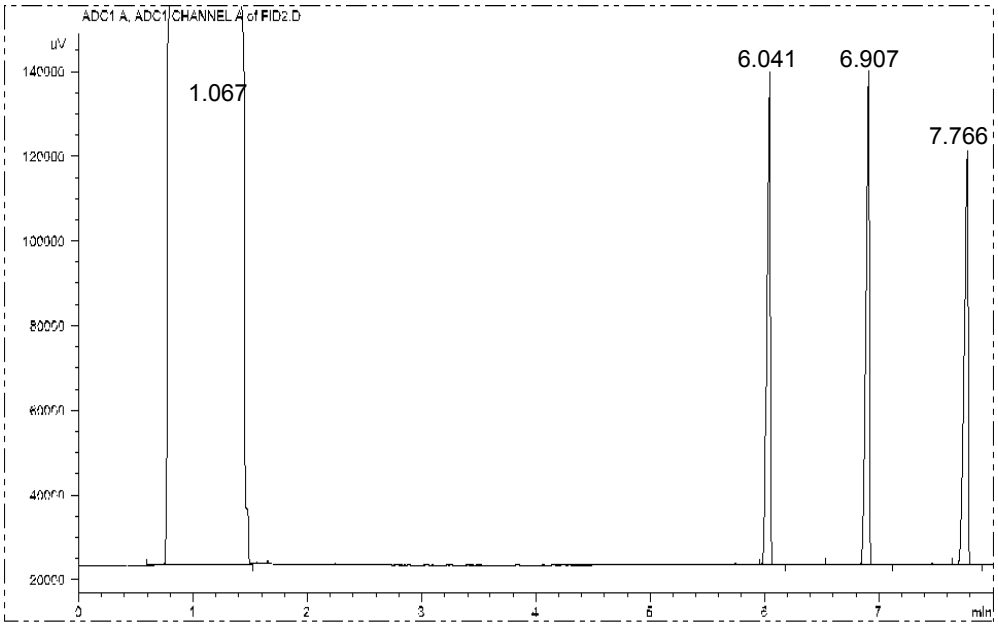
Detector

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

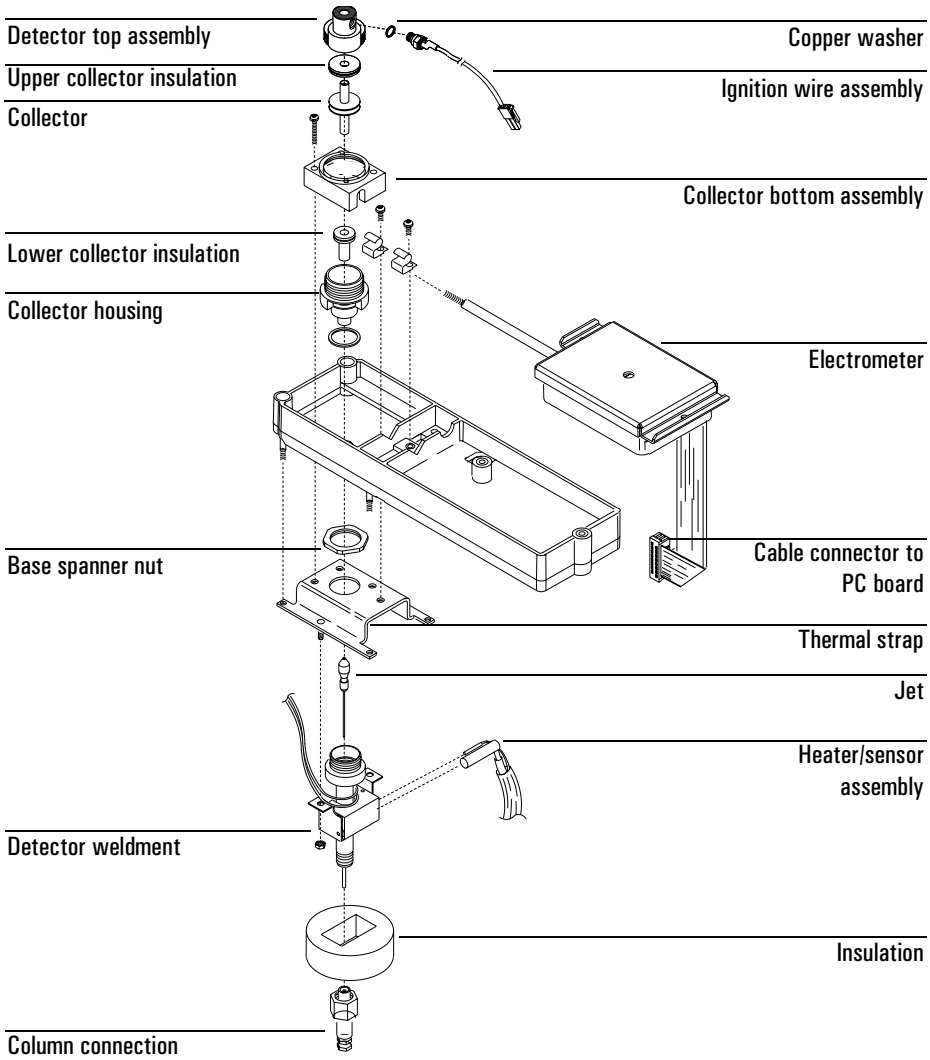
Typical FID checkout chromatogram



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

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 517.
- 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.

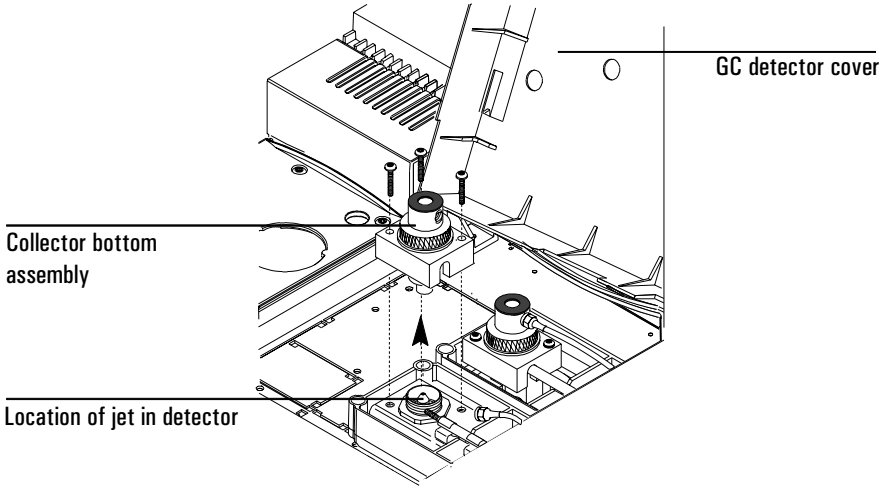
Procedure: Removing and inspecting the jet

Materials needed:

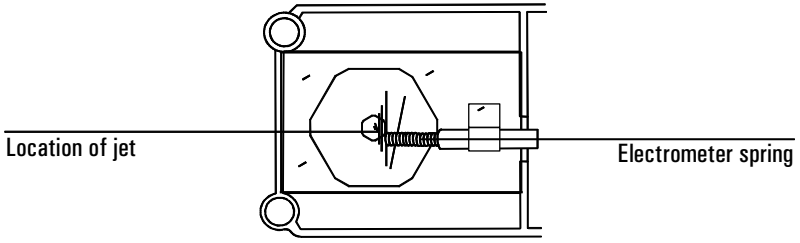
- Gloves to protect hands if detector is hot
- T-20 Torx screwdriver
- 1/4-inch nut driver
- Forceps (or tweezers)

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 "Columns and Traps" .
   - Open the GC detector cover to access the FID.

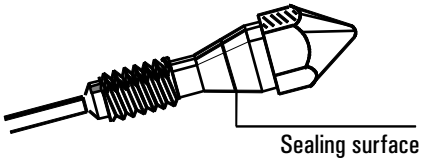
2. Put the gloves on if the detector is hot. Remove the three screws holding the collector bottom assembly in place. Lift off the assembly. The insulator can remain in the collector bottom.



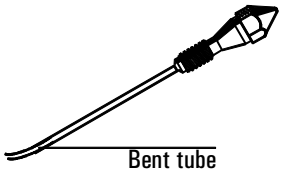
3. Using the nut driver, loosen the jet, and pull it straight out. You may need to use the forceps to grasp the jet.



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.

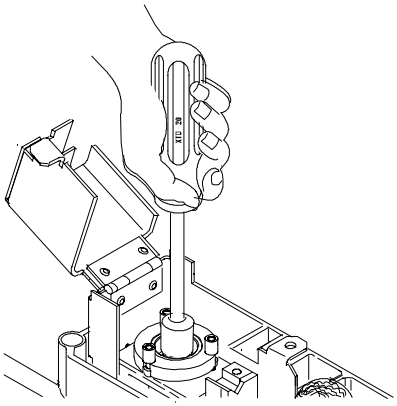
Caution Handle the clean or new jet only with forceps!

Materials needed:

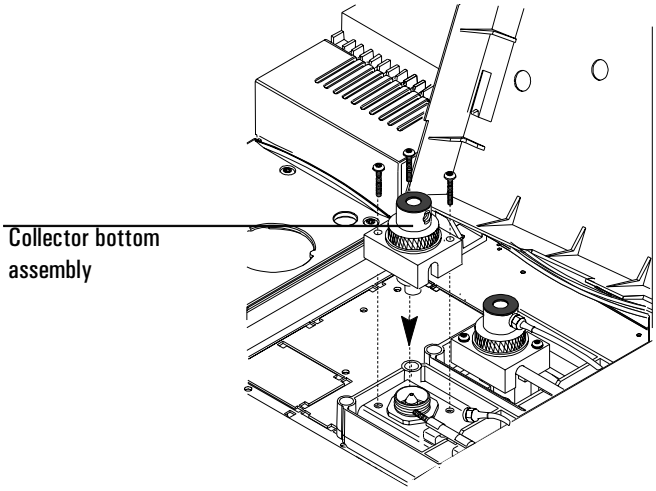
- Gloves to protect hands if detector is hot
- Forceps
- 1/4-inch hex driver
- T-20 Torx screwdriver

See page 517 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.

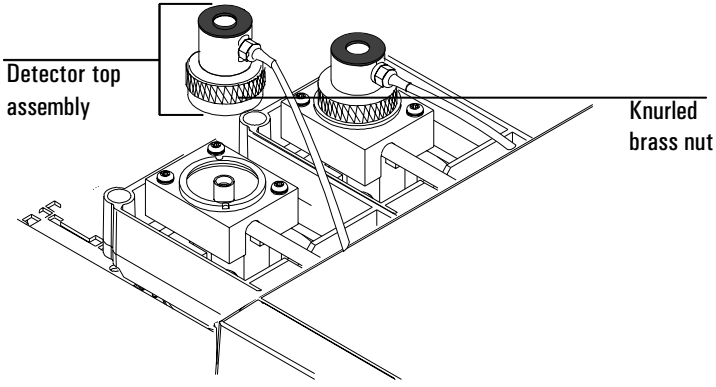
This procedure is divided into three steps: removing the collector, cleaning the collector, and reassembling the detector.

Procedure: Removing the collector

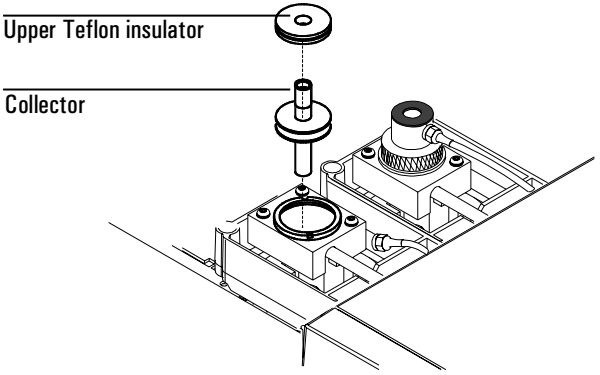
Materials needed:

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

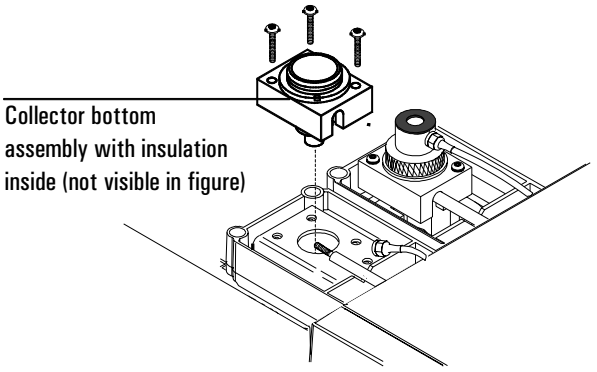
1. Complete the following preliminary steps:
   - Cool the detector to room temperature.
   - When the detector is cool, turn off the temperature zone and the gases at the GC keyboard.
   - Turn off the electrometer; the electrometer control is in the Config Det table. Press [Config] [Front Det] or [Config] [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.

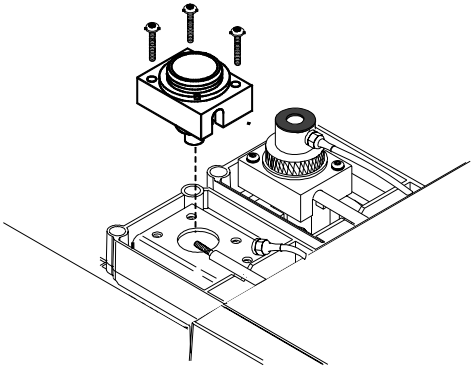
Procedure: Reassembling the detector

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

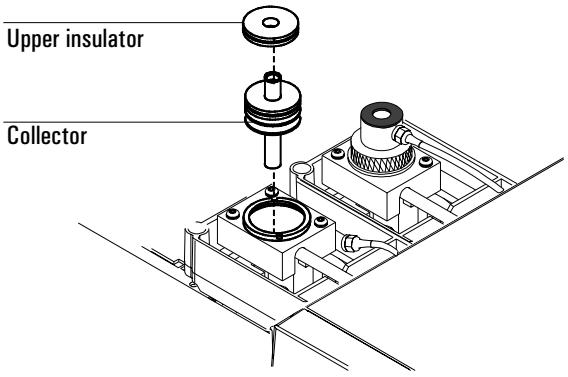
Materials needed:

- Forceps or tweezers
- T-20 Torx screwdriver

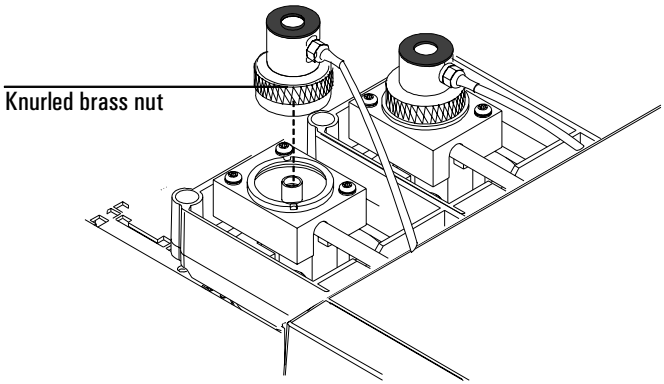
1. Insert the lower insulator into the lower collector assembly. Install the lower collector assembly and tighten the three screws.



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



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



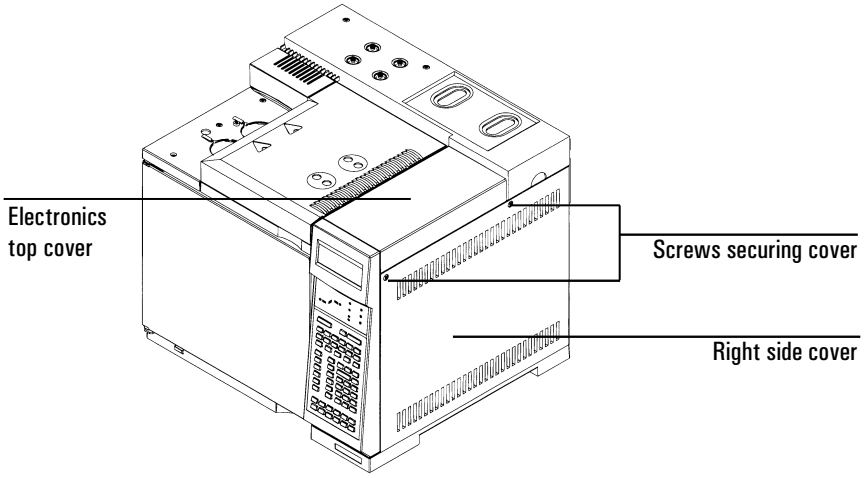
4. Close the GC detector cover. You can now restore normal operating conditions.

Procedure: Replacing the FID ignition wire

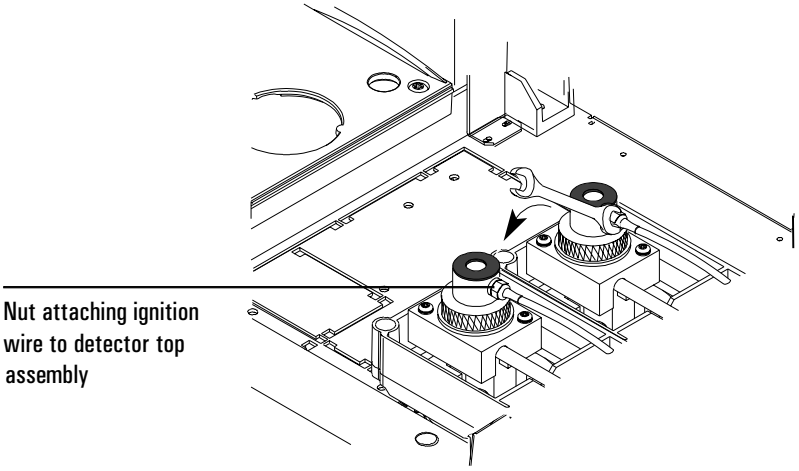
Materials needed:

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

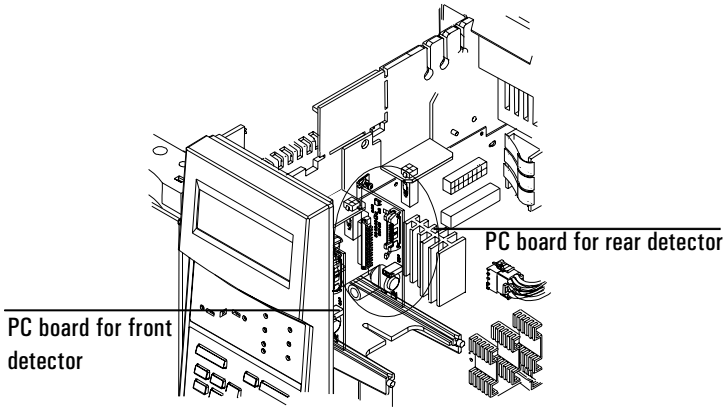
1. Complete the following preliminary steps:
   - Allow the detector to cool to room temperature. When the detector is cool, turn off the GC.
   - Lift the GC detector cover to access the FID.
   - Remove the electronics top cover.
2. Remove the two screws securing the right side cover and remove the cover.



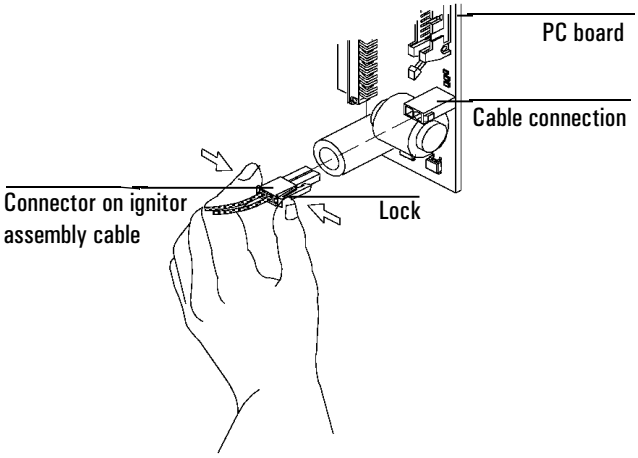
3. Using the wrench, loosen the ignition wire from the detector top assembly. Disconnect the wire completely. Do not lose the small copper washer between the top assembly and the ignition wire connection.



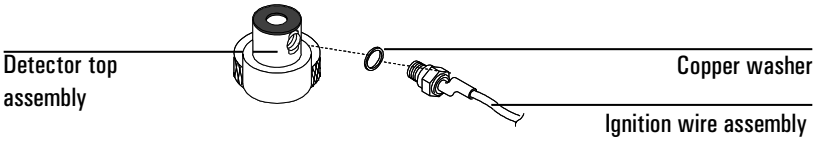
4. The other end of the ignition cable is connected to the detector PC board. Use the figure below to locate the PCB. Make sure to put on the ESD wrist strap at this time and connect it to a proper ground.



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.

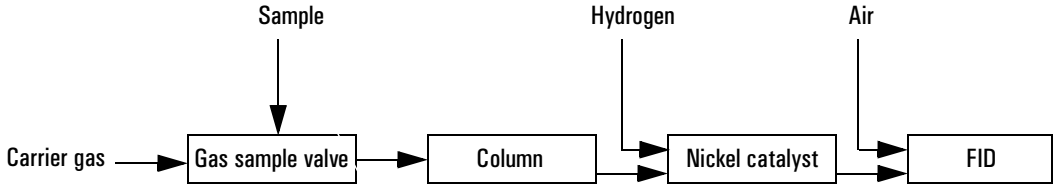


7. Replace the right side cover and the two screws. Replace the electronics top cover.

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

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 "Procedure: Measuring gas flows with a bubble meter".

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 tube375°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.

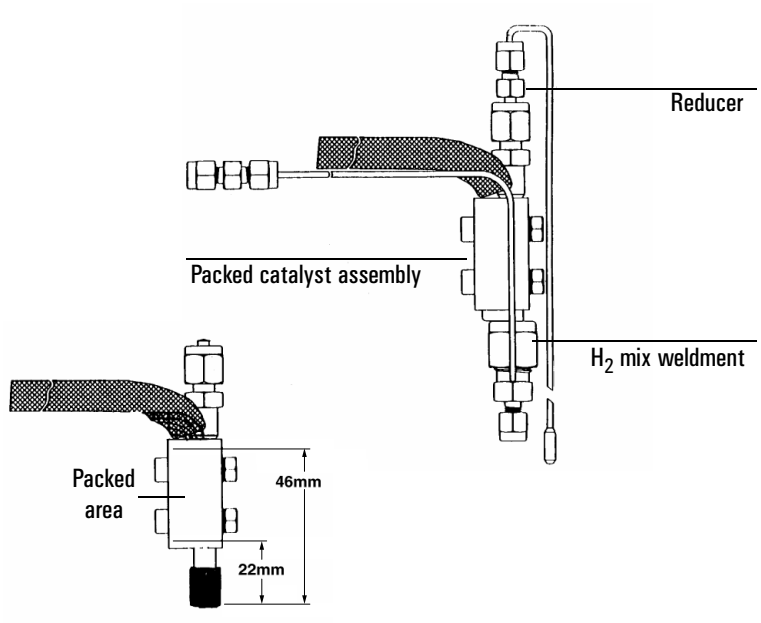
WARNING Both nickel oxide and some forms of silicon oxide are considered carcinogens for humans. Perform all work in a fume hood and wear cotton gloves at all times. Remove any spills with an HEPA-type vacuum cleaner, avoiding any action that raises dust. Alert your company's Safety group if a spill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