

Installation and Operating Guide
Nickel Catalyst Tube
Accessory 19205A

© Agilent Technologies 2000

All Rights Reserved. Reproduction, adaptation, or translation without permission is prohibited, except as allowed under the copyright laws.

Part number 19205-90107

First Edition, DEC 2000

Replaces Part No.19205-90100 Operating and Service Manual.

HP® is a registered trademark of Hewlett-Packard Co.

Printed in USA

Safety Information

The Agilent Technologies Nickel Catalyst Tube 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 Agilent 19205 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.

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.

Sound Emission Certification for Federal Republic of Germany

Sound pressure $L_p < 68$ dB(A)

During normal operation

At the operator position

According to ISO 7779 (Type Test)

Schallemission

Schalldruckpegel $LP < 68$ dB(A)

Am Arbeitsplatz

Normaler Betrieb

Nach DIN 45635 T. 19 (Typprüfung)

The Nickel Catalyst Tube Accessory 19205A

The 19205A Nickel Catalyst Tube Accessory is used on the 4890/5890 gas chromatographs for trace analysis of CO and CO₂ using a Flame Ionization Detector (FID). The gas sample is separated on the column, then passes over the hot catalyst in the presence of hydrogen so that the CO and CO₂ are converted to CH₄.

The Nickel Catalyst Tube Accessory is installed in the inlet B position. These instructions describe installation of the accessory for use with a gas sampling valve (although the accessory can be used with any inlet).

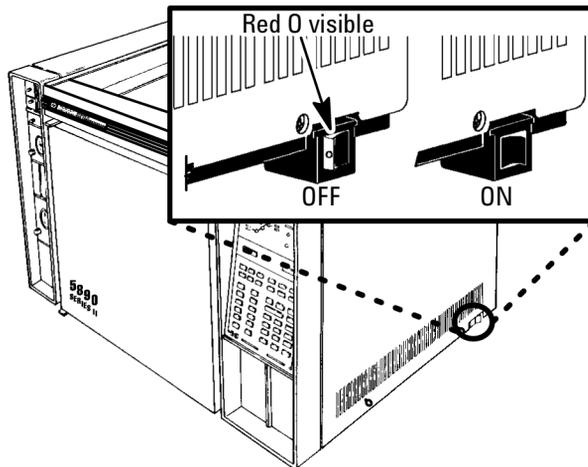
Prepare the Instrument

WARNING

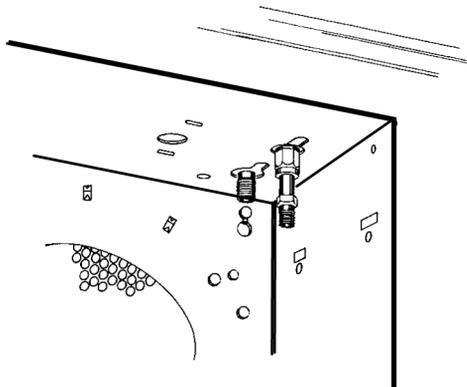
Hazardous voltages are present in the instrument whenever the power cord is connected. Avoid a potentially dangerous shock hazard by disconnecting the power cord before working on the instrument.

The Nickel Catalyst Tube Accessory 19205A
Prepare the Instrument

1. Set the main power line switch to the off position.

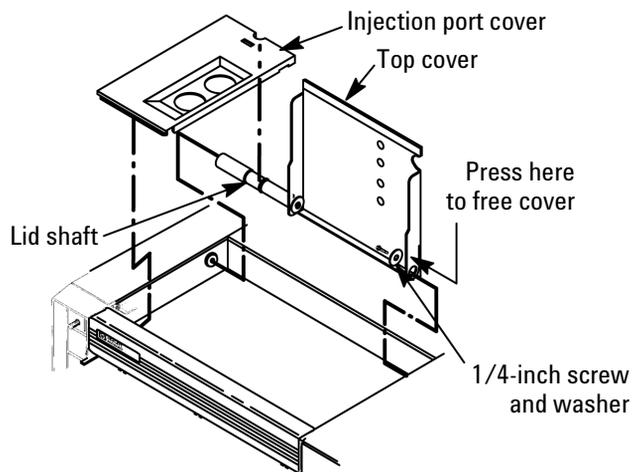


2. Disconnect the power cable from its receptacle.
3. Allow time for the oven and heated zones to cool.
4. When the heated zones are cool, turn off all gas supplies at the source.
5. Remove the column and any hardware attached to the FID (liner, column/liner nuts, ferrules, makeup gas adapter, etc.).



The Nickel Catalyst Tube Accessory 19205A
Prepare the Instrument

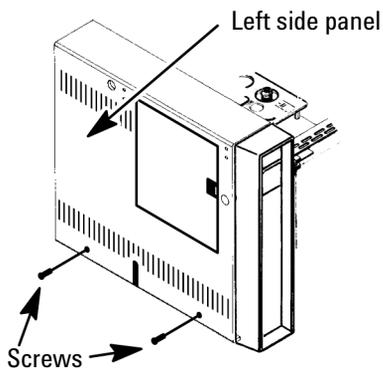
6. Remove the injection port cover by grasping its back edge and lifting it upward. If an autosampler is installed, the injection port cover will not be present.



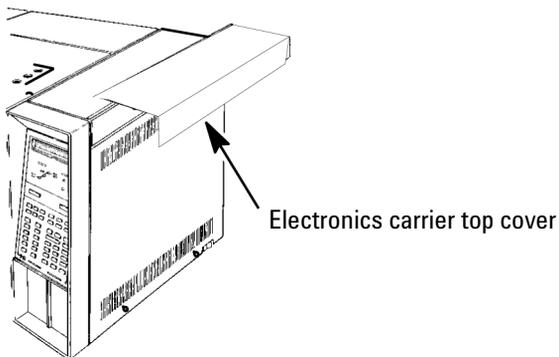
7. If an autosampler is installed on the instrument, it will be necessary to remove it and its mounting bracket to allow removal of the left side cover.
 - a. Remove the autosampler tray from its mounting bracket by simultaneously lifting and turning the two tray locks that hold it in position, then sliding the tray away from the instrument.
 - b. Lift the autosampler tray from its mounting bracket and set it aside.

The Nickel Catalyst Tube Accessory 19205A
Prepare the Instrument

- c. Remove the autosampler bracket by removing the six screws securing it to the instrument.

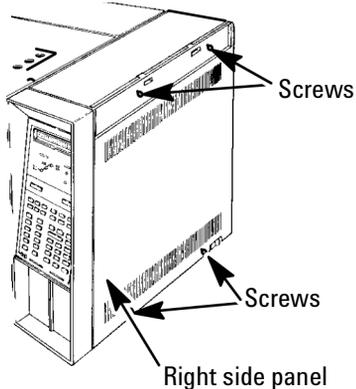


8. Remove the two screws securing the left side panel along its bottom edge.
9. Slide the left side panel towards the rear of the instrument and lift.
10. Remove the electronics carrier top cover by grasping it at the rear and lifting upwards until its catch releases, then pull it towards the rear of the instrument.



The Nickel Catalyst Tube Accessory 19205A
Install the Nickel Catalyst Tube

11. Remove the right side panel by removing four screws: two each along its top and bottom edges.



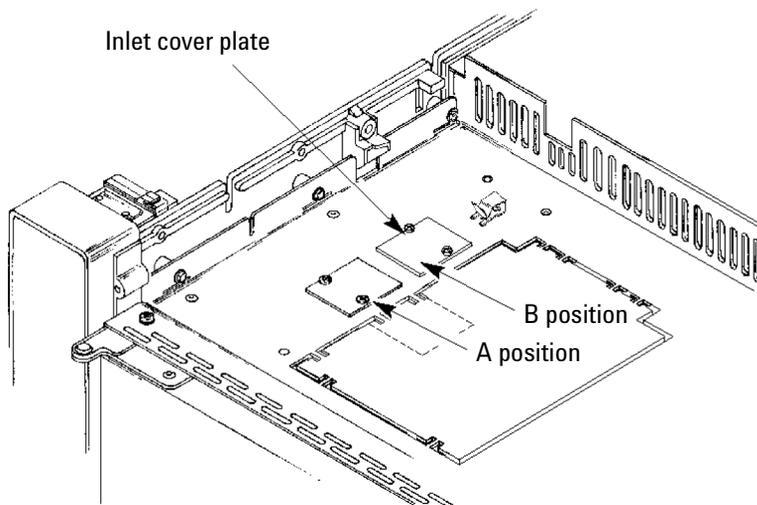
Install the Nickel Catalyst Tube

Caution

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

The Nickel Catalyst Tube Accessory 19205A
Install the Nickel Catalyst Tube

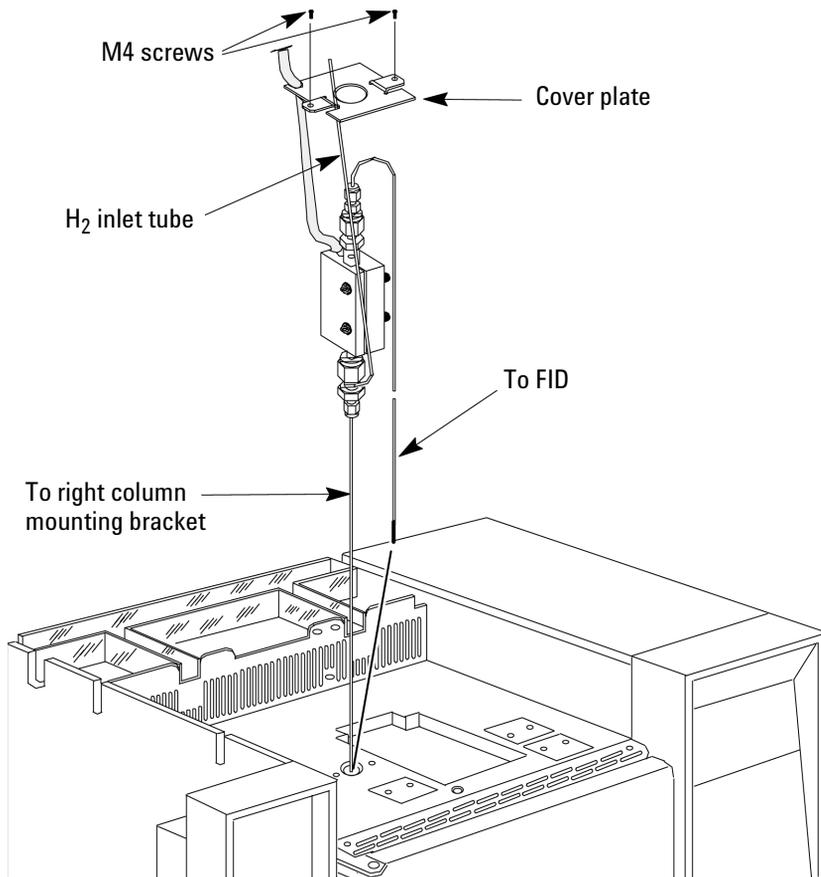
1. If present, remove the cover plate from the B inlet mounting location and the pre-cut insulation below it.



2. Using the next figure as a guide, install the Nickel Catalyst tube from the top of the oven. Use the insulation removed (tear apart as needed) to fill in the area around the Nickel Catalyst Tube.

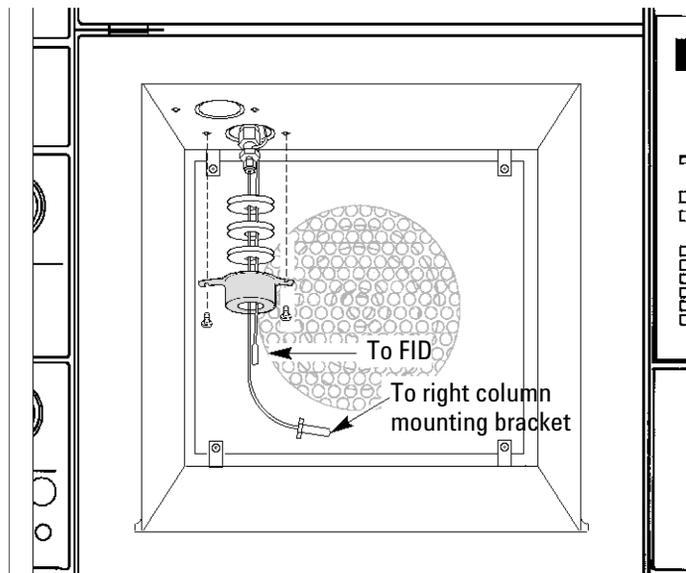
The Nickel Catalyst Tube Accessory 19205A
Install the Nickel Catalyst Tube

3. Install the cover plate provided. Secure the cover plate with two M4 screws.



The Nickel Catalyst Tube Accessory 19205A
Install the Nickel Catalyst Tube

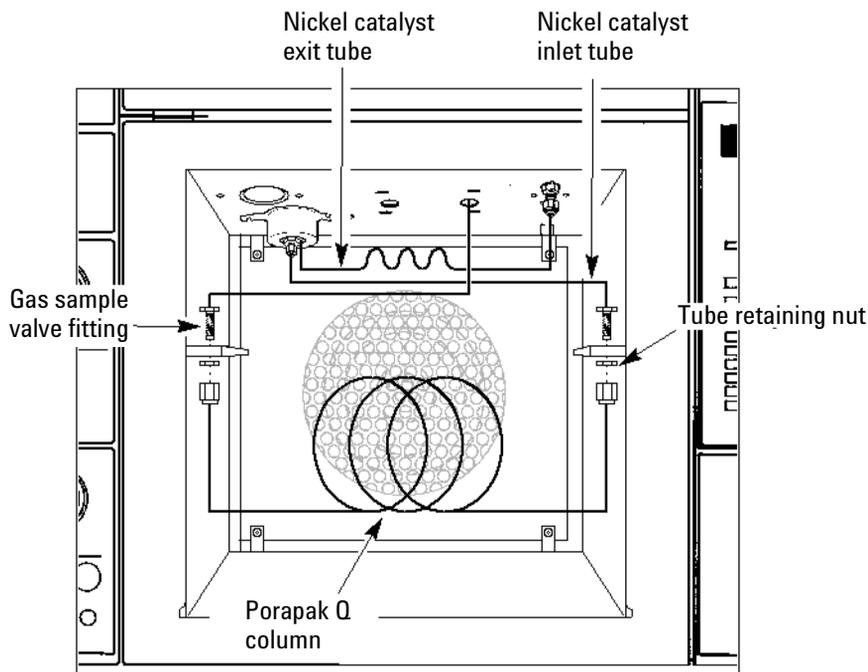
4. Place the three pieces of insulation in the insulation cup. Install the cup over the carrier gas tubing and nickel catalyst tube inside the oven. Secure the cup using two M4 screws provided.



5. Connect the nickel catalyst exit tube to the FID inlet. Assemble a 1/8-inch stainless steel nut and front and back ferrule onto the tubing. Fully insert the tube, ferrules, and nut onto the FID base. Tighten the

The Nickel Catalyst Tube Accessory 19205A
Install the Nickel Catalyst Tube

nut finger-tight. Use a wrench to tighten the nut an additional 1/2 to 3/4 turn.



6. Install the nickel catalyst inlet tube to the column bracket on the right side of the oven.
7. Install the 6-ft. Porapak Q column between the gas sampling valve fitting on the inlet side of the oven and the nickel catalyst inlet tube on the detector side of the oven.

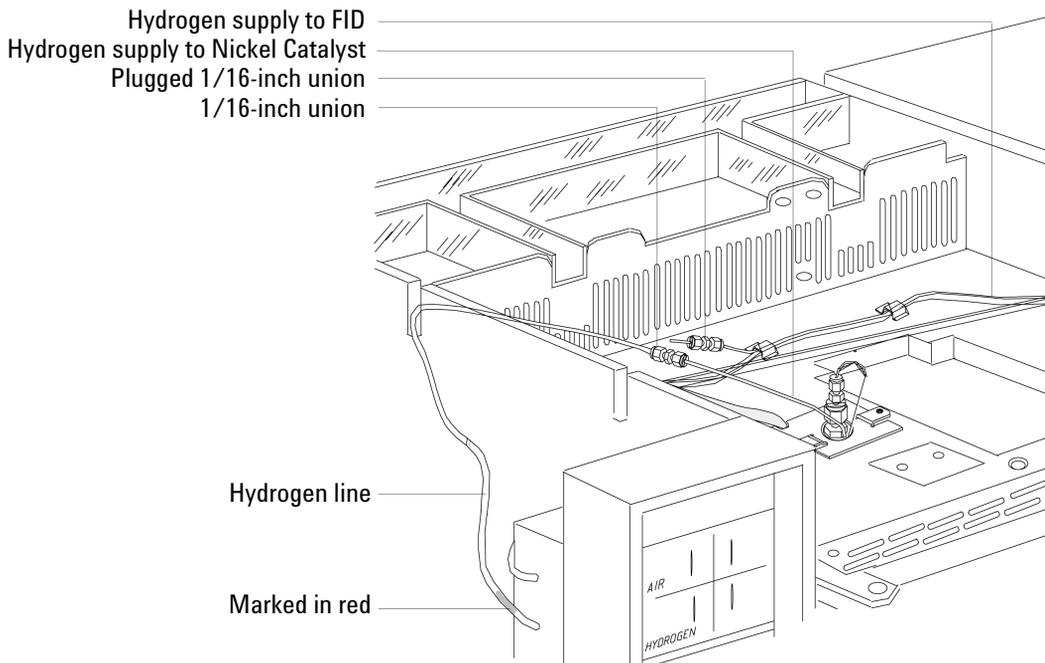
WARNING

Hydrogen (H₂) is flammable and is an explosion hazard when confined in an enclosed space (for example, the oven). In any application using H₂, turn off the supply at its source before working on the instrument.

8. Turn off the hydrogen at its source. Locate the hydrogen line (marked in red) at the flow manifold that feeds the FID being used. Trace the line to the area behind the nickel catalyst tube. Using a suitable tubing

The Nickel Catalyst Tube Accessory 19205A
Install the Nickel Catalyst Tube

cutting tool, carefully cut the hydrogen line. Be careful not to crush the tubing while cutting it.



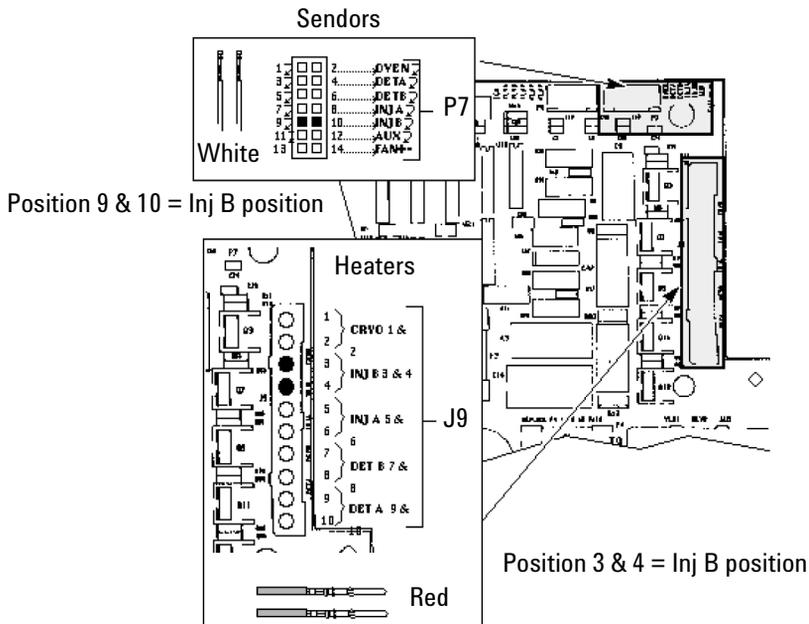
9. Using the stainless steel 1/16-inch union provided, connect the hydrogen supply side of the line to one side of the union. Insert the tube into the fitting fully. Use a wrench to tighten the nut 1/2 to 3/4 turn past finger-tight.
10. Insert the 1/16-inch hydrogen supply tube from the nickel catalyst tube into the other end of the union and tighten as described above.
11. Install the plugged union provided on the hydrogen line from the FID detector.

The Nickel Catalyst Tube Accessory 19205A
Install the Nickel Catalyst Tube

- Route the heater and sensor wires through the cable tray behind the oven to the main circuit board. Install the pins in the B injection port connector positions.

4890 Series and 5890 SERIES II Main Circuit Board

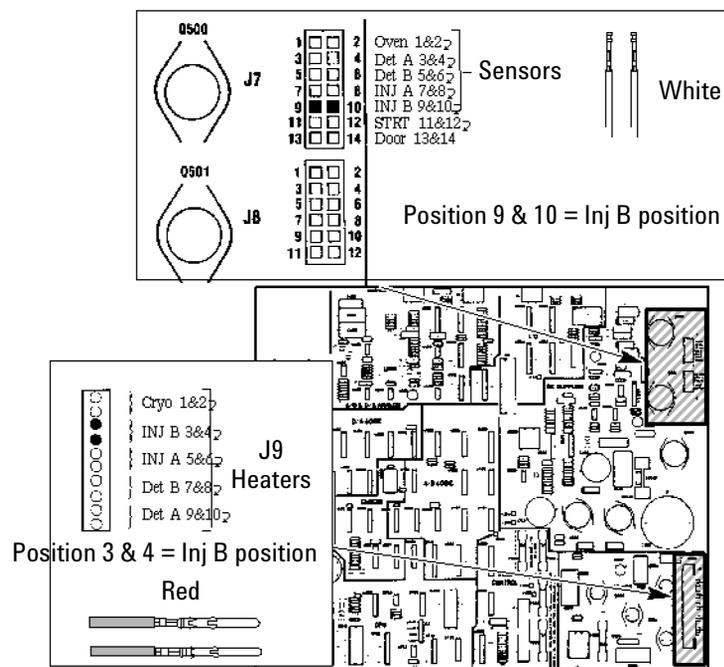
Connect the sensor leads (white) to P7, heater leads (red) to J9.



The Nickel Catalyst Tube Accessory 19205A
Install the Nickel Catalyst Tube

5890A Main Circuit Board

Connect the sensor leads (white) to J7, heater leads (red) to J9.



WARNING

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

13. Restore carrier gas flow and leak test the new installation.
14. Reinstall the panels and covers. Reconnect the instrument power cord and restore power.

Operation

Operation

Verifying Performance

1. If any heated zone is turned on, turn it off and allow it to cool. DO NOT heat any heated zone.
2. Set the gas flows.

Table 1. For a standard FID installation:

Flow	mL/min
Carrier flow (helium)	30
FID hydrogen flow	30
FID air flow	400

Table 2. If there is a TCD/FID in series installation:

Flow	mL/min
Carrier flow (helium)	30
TCD switching flow	25
FID hydrogen flow	45
FID air flow	500

3. Allow helium and hydrogen to flow through the system for 15 minutes.
4. Carefully check the column and supply fittings for leaks. Air leaks can damage the catalyst.
5. Condition the nickel catalyst by setting the nickel catalyst tube temperature (INJ B TEMP) to 375°C and the FID temperature to 400°C. Allow them to condition for 1 hour. While the zones are heating, ignite the FID flame.

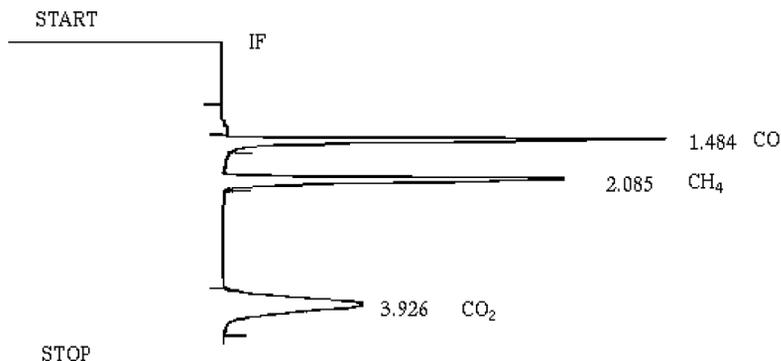
Operation

6. After 1 hour, set the following temperature:

Setting	°C
Oven temperature	35
FID temperature	as desired
Nickel catalyst	375
TCD (if in use)	as desired

When the temperatures have stabilized, the system is ready for use.

7. Load the sample loop with the nickel catalyst test mixture (a quick press of the can button) and start the run. The chromatogram should be similar to the one in the figure below (CO₂ may be larger due to the presence of air in the sample.)

**Using the Nickel Catalyst**

To use the Nickel Catalyst accessory most efficiently, keep the following in mind:

- Avoid exposing the catalyst to oxygen, which can damage it.
- If performance is significantly degraded, repack the catalyst tube.

Maintenance

Repacking the Catalyst

Should it be necessary to repack the nickel catalyst tube because of loss of conversion efficiency, repack the catalyst as described below.

WARNING

Hydrogen (H₂) is flammable and is an explosion hazard when confined in an enclosed space (for example, the oven). In any application using H₂, 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. It is important to perform all work in a fume hood and to wear cotton gloves at all times. Remove any spills with a HEPA type vacuum cleaner, avoiding any action that creates dust. Alert your company's safety group when a spill is created.

WARNING

Due to the possibility of dermatitis, wash the arms and hands with soap and water after use. The use of long sleeves is required during any use and spill cleanup. If long sleeves are not worn, long gloves are an acceptable substitute. After the assembly is filled with catalyst, the whole assembly should be carefully wiped to remove any catalyst dust.

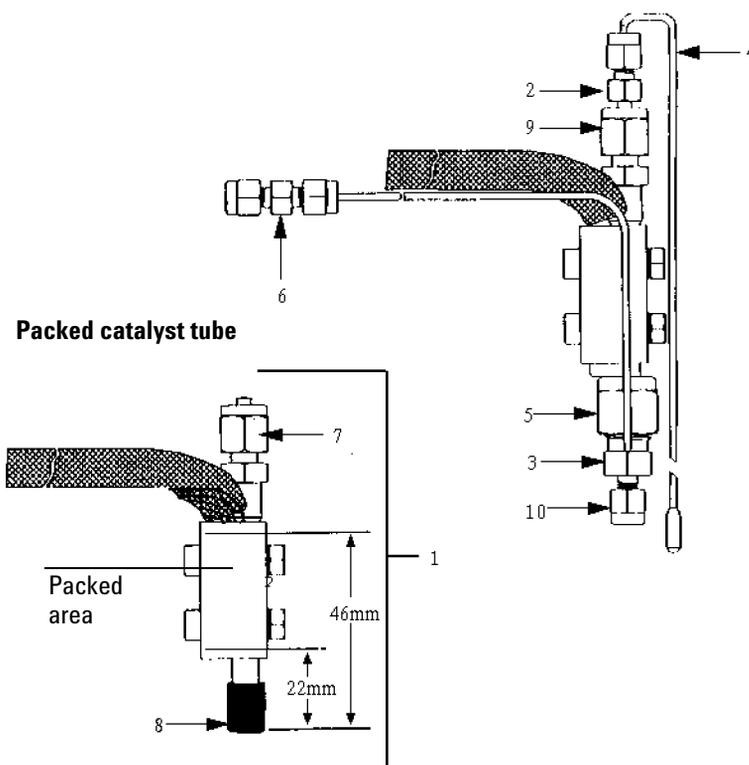
Caution

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

1. Turn off the INJ B TEMP. Turn off all other heaters. When the nickel catalyst tube is cooled to room temperature, turn off the power to the GC and disconnect the power cord. Bleed down the hydrogen and carrier gas.
2. Use a Pozidrive screwdriver to remove the two screws holding the injector cover plate on top of the nickel catalyst tube. Remove the plate and the insulation around the nickel catalyst tube.

Maintenance

3. From inside the oven, loosen the two screws holding the capillary retainer cup, rotate the cup and allow it to drop away from the base of the nickel catalyst assembly.
4. Refer to the following figure which shows the nickel catalyst assembly and the packed catalyst assembly. Use two wrenches to disconnect the H₂ mix weldment (5) from the bottom of the packed catalyst assembly (1). Be very careful NOT to place stress on the 1/16-inch tube. Stress can damage the weldment.



5. Use two wrenches to disconnect the reducer (2) from the top of the packed catalyst assembly (1).
6. Gently lift the packed catalyst assembly (1) out of the injection area. Both ends of the catalyst tube are now accessible.

7. With a hooked instrument of some type, carefully 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. Using a thin rod, 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 assure cleanliness. Dry the tube.

WARNING

The figure above shows the dimensions for repacking the tube correctly. If any catalyst is outside the heated zone, severe tailing of CO will result.

11. Prepare a simple depth gage using a wooden cotton swab or any other handy rod or tubing. Use tape or paint to mark the stick at 46 mm and at 22 mm from the blunt end.
12. Roll up a piece of glass wool into a ball 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 guage—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 nickel catalyst assembly upside down and add catalyst slowly. Tap gently on the tube to help seat it properly. 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 of the nickel catalyst is the reverse of steps 1 through 6.

Maintenance

WARNING Make sure that the insulation is carefully repacked around the tube before you reinstall the injector cover plate and the capillary retainer cup.

16. Leak test the new installation.

WARNING Hydrogen (H₂) is flammable and is an explosion hazard when confined in an enclosed space (for example, the oven).

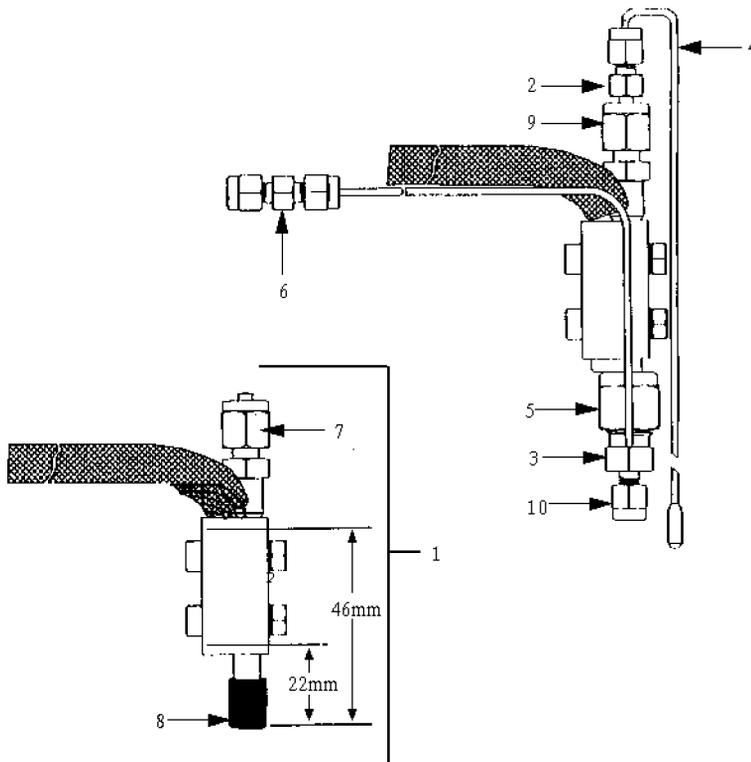
17. Start the carrier and hydrogen flows, then allow them to flow for 15 minutes.

18. Heat the nickel catalyst to 375°C and hold for 30 minutes. The accessory should be ready for use.

Replacement Parts List

Ref No.	Name	Part Number
1	Packed Catalyst Tube Assembly	18900-61020
2	Reducer	0100-0073
3	H ₂ Mix Weldment	18900-80820
4	520mm 1/16-inch Tube with 1/8-inch end	1530-2167
5	Nut, Front ferrule, and Back ferrule SS 1/4-inch (kit of 20)	5080-8753
6	1/16-inch x 1/16-inch SS Union	0100-0124
7	Brass Cap seal 1/8-inch	Supplied with replacement
8	Polyethylene Cap	Supplied with replacement
9	Nut, Front ferrule, and Back ferrule SS 1/8-inch (kit of 20)	5080-8751
10	Nut, Front ferrule, and Back ferrule SS 1/16-inch (kit of 10)	5180-4149

ns	NICAT Test Sample (pressurized can)	19354-60510
ns	Injector Cover Plate	19243-00050
ns	Capillary Retainer Cup	19243-00070
ns	Insulation for Cup (3 required)	19243-00067
ns	Column Bulkhead Fitting and Tube	07675-80050
ns	Retaining Nut for Bulkhead Fitting	2950-0203
ns	6-ft x 1/8-inch OD Porapak Q Column	19006-80015



The Nickel Catalyst Tube Accessory 19205A
Maintenance



Agilent Technologies



Printed on recycled paper.



This product is recyclable.

Agilent Technologies, Inc.
Printed in USA Dec 2000



19205-90107