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The Nitrogen-Phosphorus Detector

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
6890 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 82 shows the flow paths for the NPD.



Figure 82. NPD pneumatics

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].

FRONT DET (N	PD)
Adjust offset	0 f f
Output	0.3
Bead voltage	0 f f

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:

FRONT	DET	(NPD)
Adjust	off	set	30
Output			0.3
Bead vo	ltage	e	wait

FRO	NT DET	(NPD))
Temp	not re	ady	30
Outpu	Jt		0.3
Bead	DetTem	ip <	150

30

0.5

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 "Equilibration time") begins. The display blinks.

FRONT	DET	(NPD)	
Adjust	offs	et	30
Output			0.5
Bead vol	tage	2.	500

FRONT DET (N	NPD)
Adjusting	30
Output	9.1
Bead voltage	2.750

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.

```
      FRONT DET (NPD)

      Adjust offset
      Off

      Displays Off when finished.

      Output
      30.1

      Bead voltage
      2.850

Bead voltage does not change.
```

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 <u>"Clock time programming"</u>.

CautionIt 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]:

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 <u>"Run time programming"</u>.

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:

- Set the carrier gas in the Constant flow mode. Set detector makeup gas to Const makeup.
- If you 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 should be at least 15 Hz. To use fast peaks:

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

CONFIGURE SIGNAL 1 Fast peaks On < 2. Press [On].

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.

Jets and collectors

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

Туре	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	

Table 63. Jets and Collectors for the Capillary-Optimized NPD

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 on <u>"Replacing or cleaning the jet"</u>.

Table 64. Jets and Collectors for the Adaptable NPD

Туре	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.

Operating the NPD

Use the information in <u>Table 65</u> to select temperatures and flows. Choose a minimum source pressure from <u>Figure 83</u>. You must add 10 psi (69 kPa) to the source pressure on the chart.

Table 65.	Flows, Temperatures,	and Bead Information
-----------	----------------------	----------------------

Gas type	Recommended flow
Carrier gas (helium, hydrogen*, nitrogen)	<i>Capillary</i> , 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	Nitrogen: 5 to 10 mL/min
(helium, ** nitrogen)	Helium: less than 5 mL/min
Temperature (Default is 250° C	; range is ambient to 400° C)
$<$ 150 $^{\circ}$ ×C, the <code>Adjust</code> o	ffset process will not start.
325 to 335°C is recommended.	
Detector temperature should be g With higher detector temperature	reater than the highest oven ramp temperature. s, less bead heating voltage is required.
Adjust offset Default is 30 pA, s to 99 pA.	suggested operating range is 30 to 40 pA, and allowable range is 10
\geq 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, a	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 minute	S.
Bead voltage (range is 0 to 4.09 Set Equib time = 0.0 and Bead ve ignites.	15 V) Use to make minor adjustments or manually activate the bead. oltage at 2.0. Increase voltage in 0.01 volt increments until the bead

Gas pressures

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



Figure 83. Pressure/flow relationships for NPD gases (at 25°C and 1 atmosphere of pressure)

Operating with EPC

Press [Front Det] or [Back Det].

FRONT DI	ETI (NI	PD)	
emp	24	Off <	: [] []
12 flow	0.0	Off	
Air flow	0.0	Off	ļ.
1kup flow	0.0	Off	
djust off	set	Off	
Dutput		0.3	+
∃ead volta	ge	Off	+1

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, the control table will also include one of these:

```
Mode: Const makeup
Mkup flow O.O Off<
```

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** or equilibration time, press [Config][Front Det] or [Config][Back Det]:

CONFIGURE FRONT DET Mkup gas type He < Equib time 0.00 Electrometer On

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

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

	FRONT	DET	MAKEUP	GAS	Ì
	Heliun	n		<	İ
	*Nitro	gen			E
					Ĩ
L					Ĺ

e electrometer

Select the appropriate gas.

Figure 84. NPD control table

Procedure: Using the NPD

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 <u>Figure 84</u> 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 <u>511</u>). 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].

Short-cut procedure: If you (assumes correct setpoints If you

correct setpoints are stored)

 Open detector control table.
 Turn temperature On.
 Turn makeup gas On, if needed.
 Press [Det Control].

5.Press [On].

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 511), 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 non EPC 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.

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 μ L syringe. For a heated inlet, actual sample volume injected will also include an additional 0.4-0.7 μ L, 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.

COIU	inn and sampic	
	Туре	HP-5 30m \times 0.32mm \times 0.25 μm $$ PN 19091J-413 $$
	Sample	NPD Checkout 18789-60060
	Injection volume	1 <i>µ</i> L
Inlet	ţ	
	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

NPD checkout conditions

Column and sample

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





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

Maintaining a Nitrogen-Phosphorus Detector



Figure 85. 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 590, "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 86. The NPD bead assembly

CautionThe 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 <u>585</u> 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.

 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 66 lists the NPD jets.

Table 66. NPD Jets

Туре	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 <u>590</u>.
- 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.
- CautionThe 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

CautionThe 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 <u>594</u>.