340 Electron Capture Detector (ECD)

Theory of operation

The ECD is based on the phenomenon that electronegative species can react with thermal electrons present to form negatively charged ions. The loss of such electrons is related to the quantity of analyte in the sample. In order to produce capturable (low energy) thermal electrons, the carrier gas is ionized by beta particles from a radioactive source in the cell. This electron flow produces a current, which is collected and measured. When the sample molecule is introduced into the cell, electrons which would otherwise be captured at the electrode are captured by the sample, resulting in decreased current. This change is what is recorded and measured for the chromatogram.

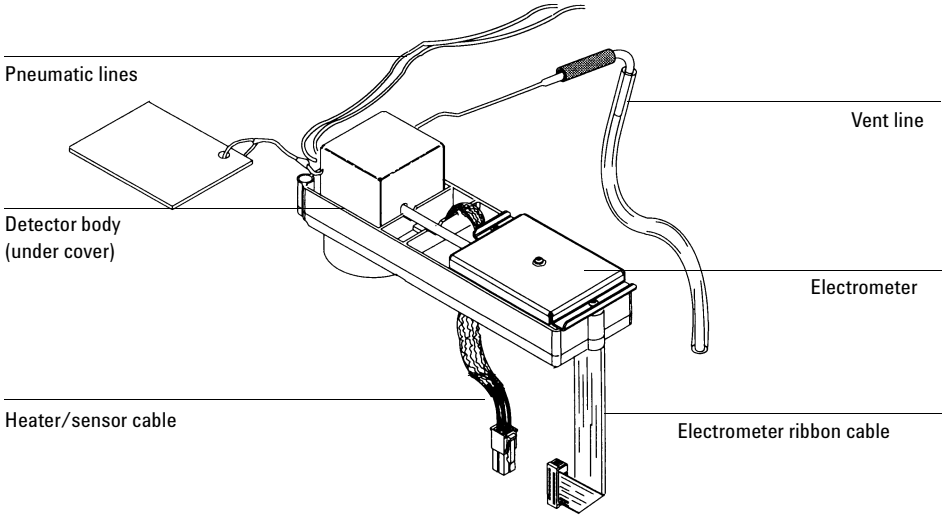

Figure 340-1 The Electron Capture Detector (ECD)

EPC detector

The EPC version of the ECD detector has one flow line for the anode purge/ makeup gas. After an initial filter frit, it splits into two paths. Each line has a proportional valve controlled by a pressure sensor, and a non-adjustable restrictor frit. The makeup gas line sweeps past the end of the column and carries the column effluent into the ECD cell. The anode purge flow sweeps the upper part of the ECD and cleans sample deposition from the detector.

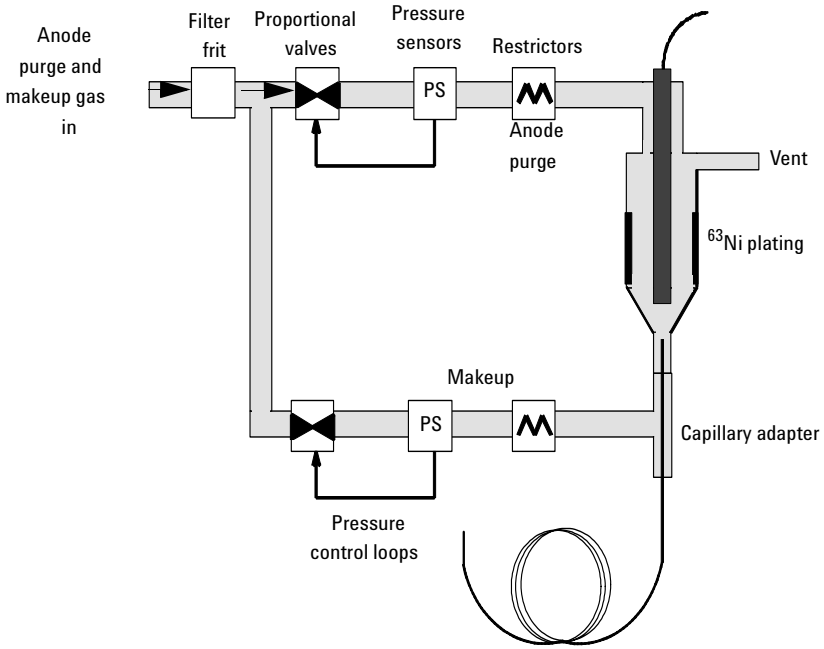

Figure 340-2 EPC ECD flow diagram

Manually controlled detector

The manually controlled ECD has a pneumatic supply line for the anode purge gas and for the makeup gas. Both lines have an on/off solenoid valve and a nonadjustable restrictor frit. A pressure regulator allows for adjusting the

makeup gas and anode purge flows. The makeup gas line sweeps past the end of the column and carries the column effluent into the ECD cell.

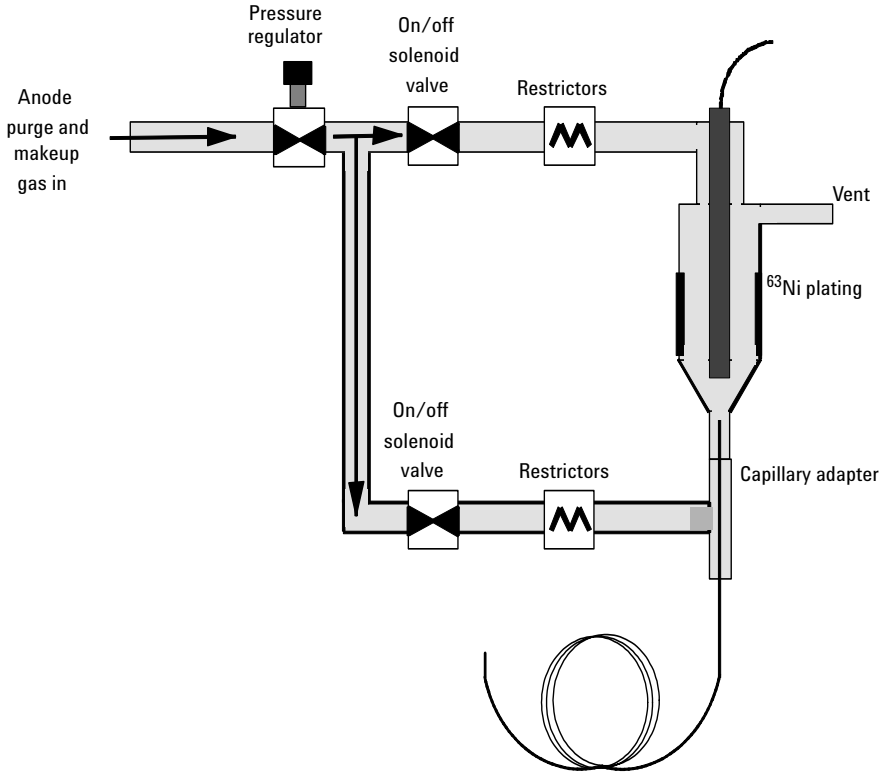

Figure 340-3 Manual ECD flow diagram

Replacement procedures

Replacing the entire detector/detector cell

WARNING Before proceeding, turn off the oven and any heated zones and let them cool down. Turn off any detector gases at their supply, then turn off the main power switch and unplug the power cord.

WARNING The ECD cell contains radioactive ^{63}Ni. To reduce the risk of exposure, wear disposable gloves while handling the ECD cell. When you are finished, dispose of the gloves and wash your hands with soap and water.

1. Remove the detector cover, the electronics carrier cover and the right side cover.

Caution Make sure you are properly grounded with an ESD strap before continuing.

2. Disconnect the electrometer ribbon cable from the ECD interface card.
3. Disconnect the heater/sensor leads from the connector on the right side of the GC.

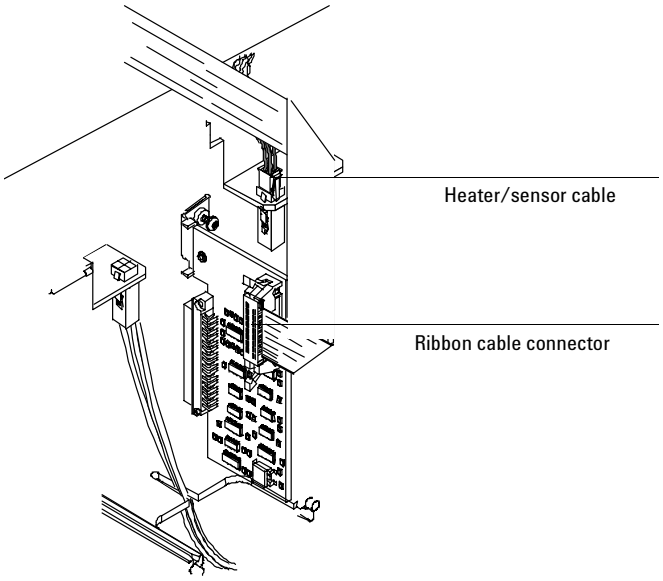

Figure 340-4 Disconnecting the ECD cables

4. Inside the oven, remove the insulation cup and disconnect the column from the makeup gas adapter.
5. Use a 9/16-inch wrench to loosen the 1/4-inch Swagelok nut on the makeup gas adapter from the bottom of the detector. Slide the makeup gas adapter out of the bottom of the detector.

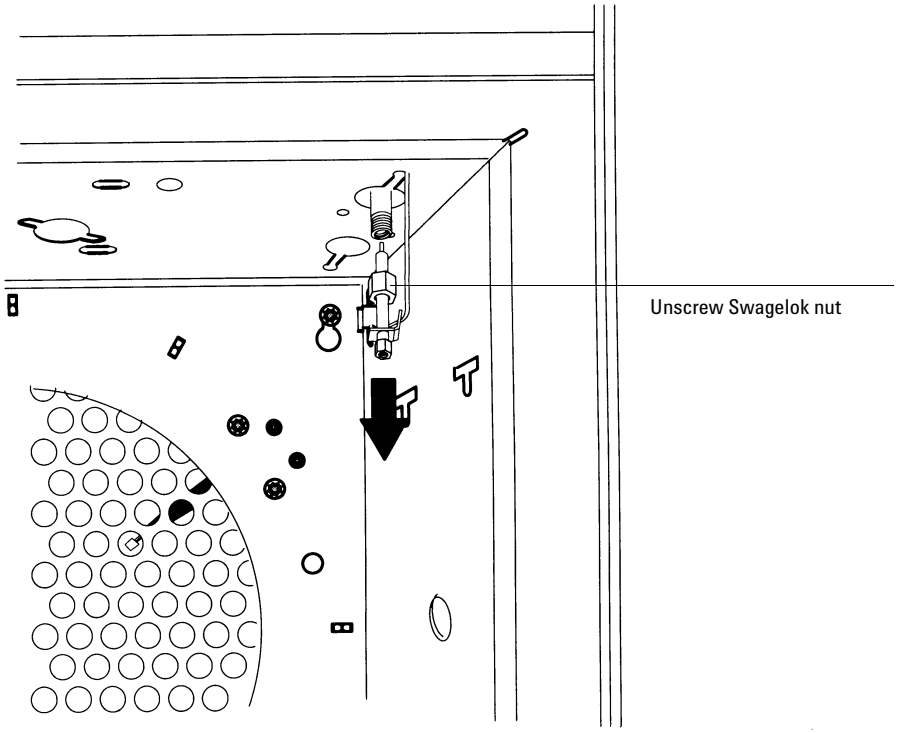

Figure 340-5 Disconnecting the makeup gas adapter

6. Disconnect the ECD vent tube at the rubber sleeve.
7. Use a 5/16-inch wrench to disconnect the 1/16-inch Swagelok union on the anode purge line.
8. Remove the one Torx T-20 screw securing the top ECD detector cover to the detector pallet and remove the cover.
9. Disconnect the anode signal wire from the detector.

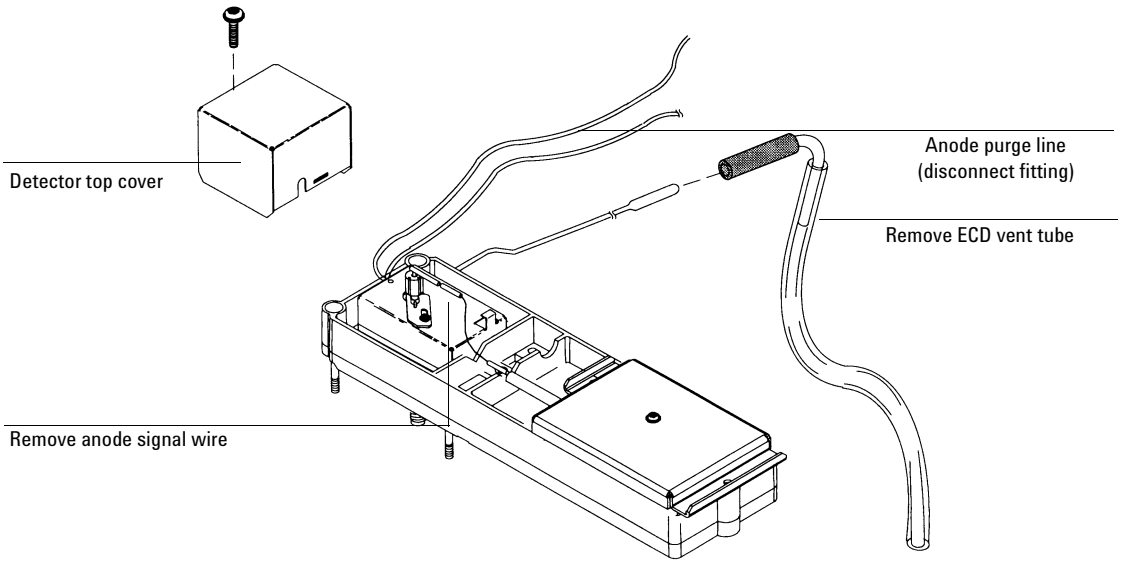

Figure 340-6 Removing the cover, anode purge fitting, vent tube, and anode signal wire

10. Fully loosen the four Torx T-20 screws on the detector pallet and lift the pallet and detector from the GC.
11. Thread the heater/sensor leads out of the detector pallet and remove the detector from the pallet.

The ECD detector is now properly disassembled for replacement or exchange. DO NOT remove the inner thermal cover if you are replacing the entire detector assembly.

Replacing the heater/sensor assembly

After removing the ECD detector from the GC, you can further disassemble it to replace the heater/sensor assembly.

WARNING The ECD cell contains radioactive ^{63}Ni. To reduce the risk of exposure, wear disposable gloves while handling the ECD cell. When you are finished, dispose of the gloves and wash your hands with soap and water.

1. Remove the detector as described in the Replacing the entire detector/ detector cell procedure in this section.
2. Loosen the locking tab screw on top of the detector, slide the locking tab back, and pivot it out of the away.

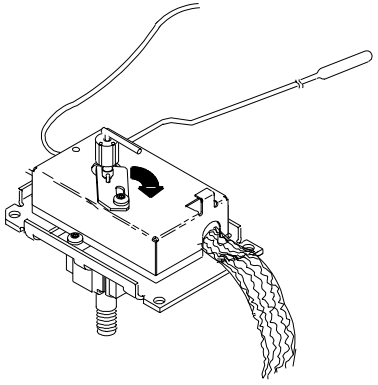

Figure 340-7 Unlocking the thermal cover

3. Lift the thermal cover up and carefully slide it off of the anode assembly.

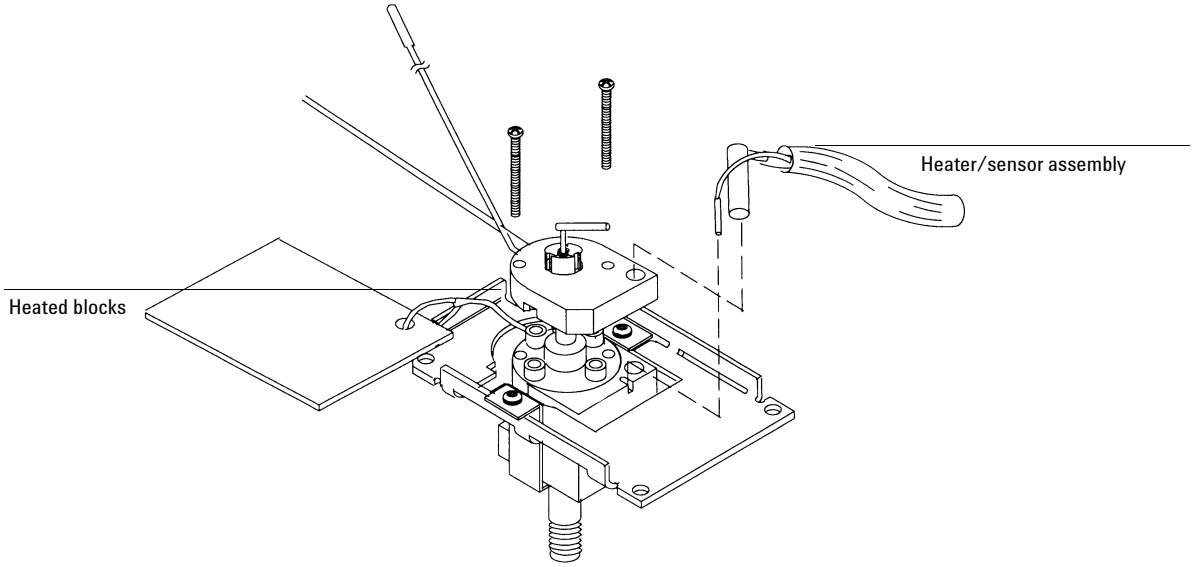

Figure 340-8 Removing the heater/sensor

4. Slide the heater and sensor out of the heated blocks.

Replacing the makeup gas adapter

After removing the ECD detector from the GC, you can further disassemble it to replace the makeup gas adapter. The makeup gas adapter consists of a line from the detector pneumatics manifold that carries makeup gas to a weldment that screws into the bottom of the ECD detector. From there, the makeup gas sweeps past the end of the column and carries the column effluent into the ECD cell.

1. Remove the detector as described in the Replacing the entire detector/ detector cell procedure in this section.
2. Remove the Torx T-20 screw holding the pneumatics block(s) to the detector manifold.

Note There are two pneumatics blocks on the EPC version of the ECD pneumatics manifold. The outside block is the anode purge gas line and the inside block is the makeup gas line.

3. Slide the makeup gas adapter up and out of the GC.
4. When reinstalling the makeup gas adapter, ensure the following:
   - Approximately 6 inches of the makeup gas line should reside in the oven after installation.
   - The makeup gas line should be bent into a coil that loops around the bottom of the detector weldment and makeup gas adapter.

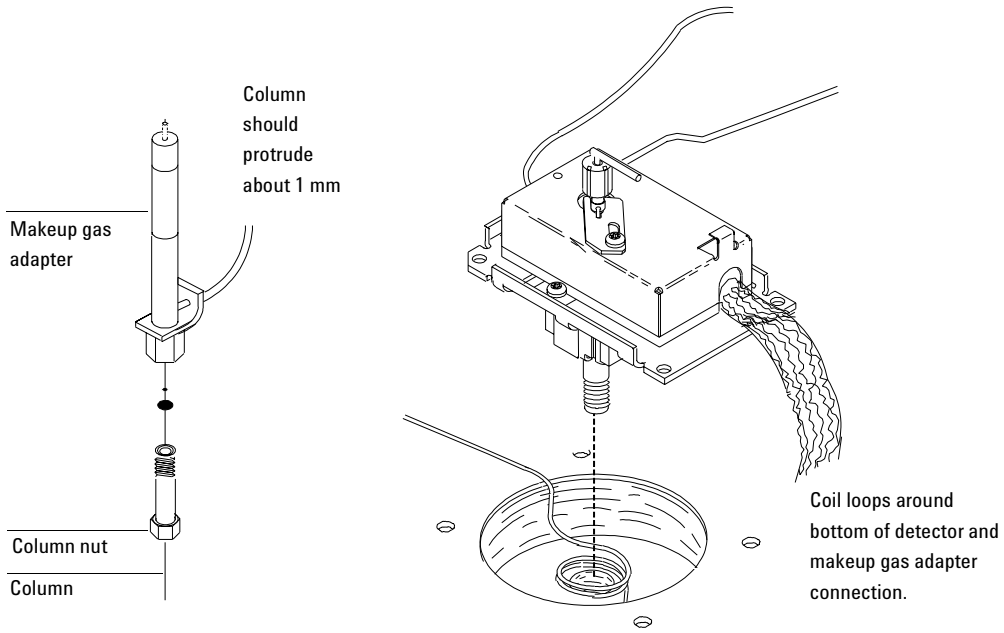

Figure 340-9 Proper configuration of the column and makeup gas tubing

- The end of the column should protrude about 1 mm from the top of the makeup gas adapter. The total dimension from the back of the column nut to the end of the column will be about 75 to 76 mm.

Removing the EPC flow manifold

The ECD detector uses a Type 1 EPC flow manifold that contains one inlet supply fitting for a purge/makeup gas.

WARNING Before proceeding, turn off the oven and any heated zones and let them cool down. Turn off any detector gases at their supply, then turn off the main power switch and unplug the power cord.

1. Remove the top plastic covers from the detector and pneumatics areas. Also remove the RFI metal shield and the top rear metal cover.

2. Remove the gas supply fitting from the side of the manifold.
3. At the front of the manifold, remove the Torx T-20 screw that holds the manifold in its slot.

Caution Make sure you are properly grounded with an ESD strap before continuing.

4. Unlock the detector manifold's ribbon cable from the EPC board and detach the connector. The adjacent ribbon cable may have to be removed as well.
5. Remove the Torx T-20 screw holding the output pneumatics block(s) to the manifold and remove the block.
6. Slide the manifold from its slot.

Note There are two pneumatics blocks on the EPC version of the ECD pneumatics manifold. The outside block is the anode purge line and the inside block is the makeup gas line.

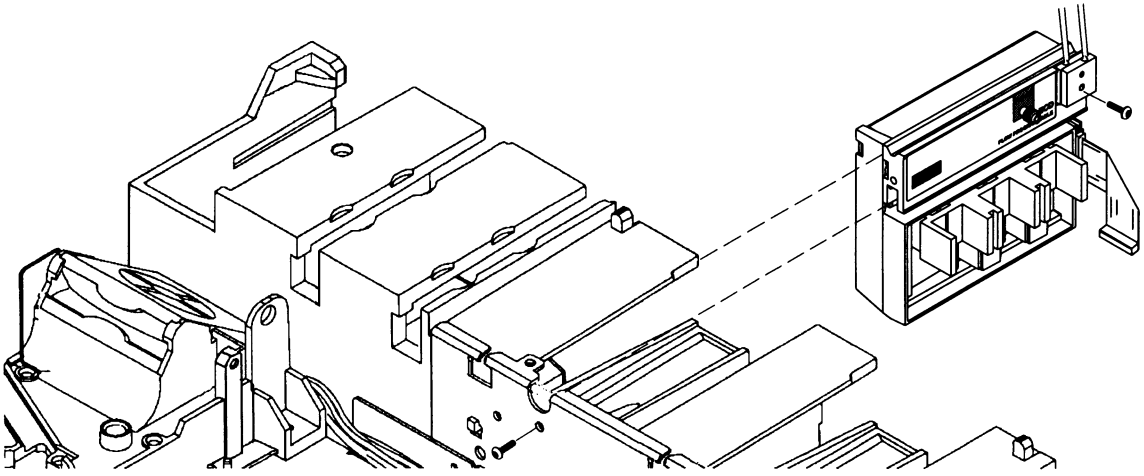

Figure 340-10 Removing the detector flow manifold

Removing the signal board

1. Remove both the electronics top cover and the right side cover.
2. Remove the Torx T-20 screw securing the top cover and remove the cover.
3. Disconnect the signal wire from the signal board interconnect.

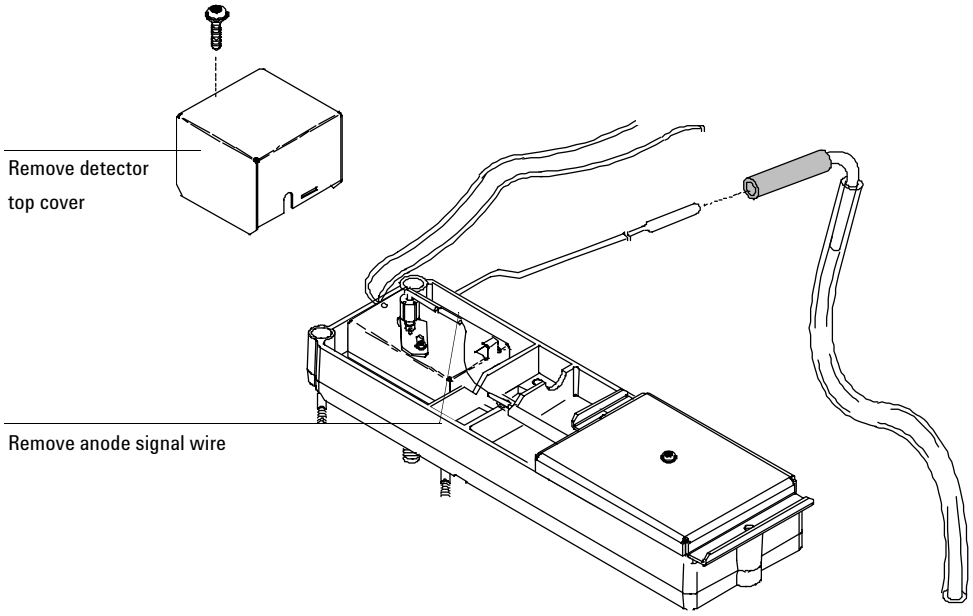

Figure 340-11 Removing the top cover and anode signal wire

4. Remove the screw and clamp on the electrical interconnect.
5. Remove one Torx T-20 screw from each end of the signal board. (Do not remove the screw on the top of the cover.)

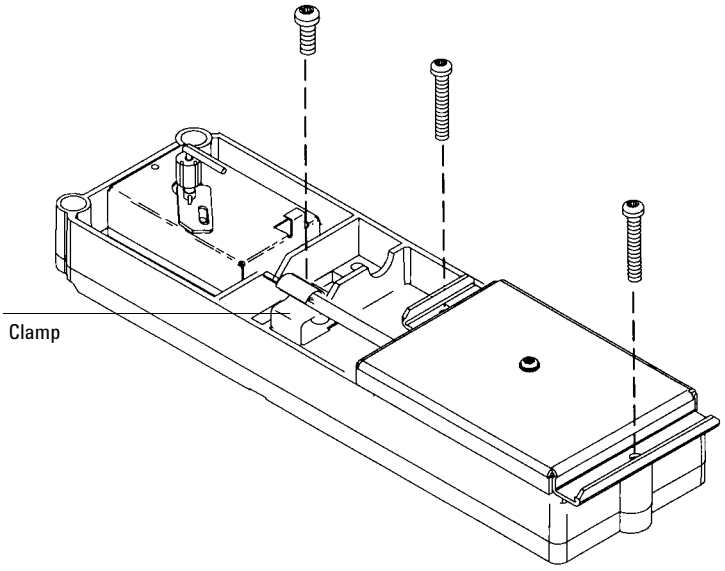

Figure 340-12 Removing the ECD signal board

6. Unlock and detach the ECD signal board's ribbon cable from the detector's interface board and lift the signal board from the detector pallet.
7. Reassembly is the reverse of removal.

Replacing the detector interface card

The ECD detector interface board plugs into the main board as shown below.

1. Unlock and unclip the ribbon cable from the interface board.
2. Remove the screw at the top of the card where it mounts to the main board and pull the card out.

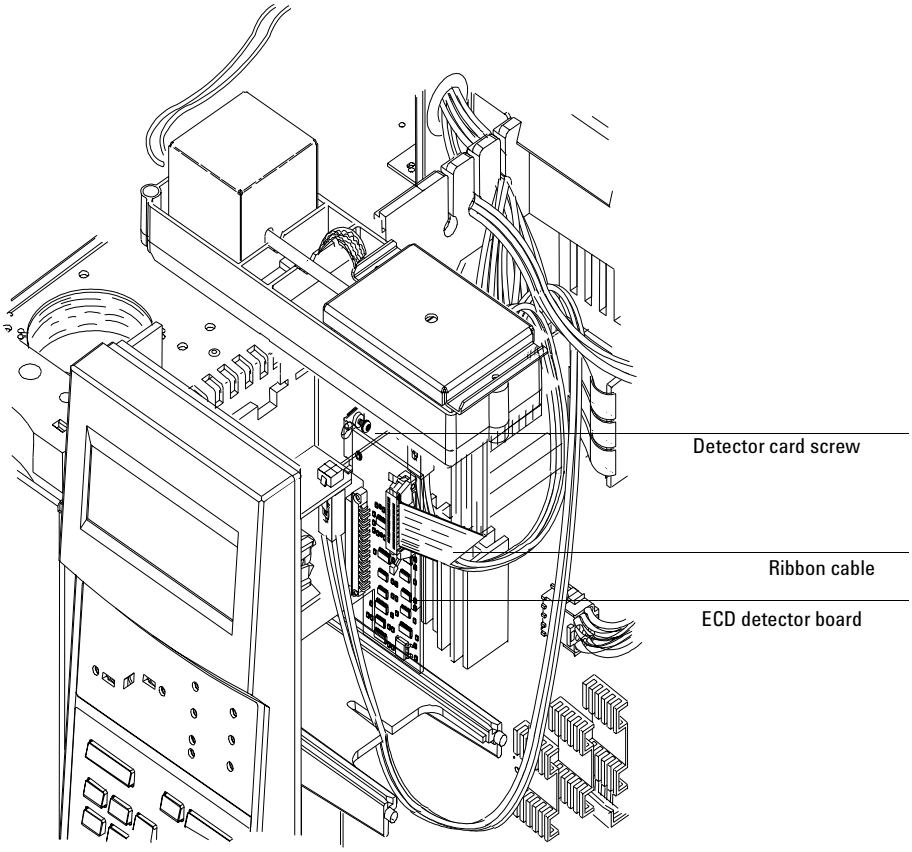

Figure 340-13 Detector interface board installed in back detector position

Diagnostics

Frequency test

Perform this test to make sure that the base frequency for the ECD during a blank run indicates a relatively contaminant-free system.

Note It may take 24 hours for the ECD baseline to completely stabilize, especially if you are starting with a cold system and want to assure high-sensitivity operation.

Therefore, for the most accurate results, run the detector at normal operating conditions for as long as possible (at least 2 hours and up to 24 hours) before running the frequency test.

If you will be injecting into an unused inlet, you must use low-bleed septa. Make sure to condition new septa before use in an inlet for several hours with 1 to 5 mL/min carrier flow.

1. Make sure you are using normal operating conditions and that *at least* two hours have elapsed since the last run.
2. Turn on the ECD and the corresponding signal.
3. Check the displayed “Output”:
   - <100 = ECD frequency is acceptable
   - ≥100 = Contaminants in system

Note Each display count equals a frequency of 5 Hertz (e.g., a display reading of 100 = 500 Hz.).

4. If the ECD frequency indicates contamination (≥100) check for the following:
   - Contaminated carrier gas trap(s) and or supply—replace carrier gas supply tank and any traps on the carrier supply line.
   - Insufficient column conditioning—fully condition the column.
   - Contaminated detector—bake out the detector.
   - Column, inlet and/or septum bleed—clean the inlet/replace the septum with a conditioned, low bleed septum.
   - Leaks—perform leak tests on both the inlet and detector systems.
   - Anode current leakage—make sure the anode contacts are clean. Make sure the anode nut is tight.

Leak test

Note Once you have determined that the flow system components upstream from the detector (gas supply tubing, inlet, column fittings) are leak free, perform the following ECD detector leak test.

1. With the GC on and operating normally, set the oven, detector, and inlet temperatures to ambient.
2. Turn off the ECD and then turn off the inlet pressure.
3. Turn off the anode and makeup gas flows.
4. Cap the ECD exhaust vent with a vent plug (part no. 5060-9055).
5. Set carrier gas pressure at the inlet corresponding to the ECD to 15 psi (103 kPa).
6. Wait until the system reaches the setpoint pressure and then turn off the pressure and monitor the actual pressure value for at least 10 minutes.

7. Check for pressure drop:

   - If the pressure stays stable or drops only 0.5 psi, you can consider the ECD leak-free.
   - If the pressure drops more than 0.5 psi, you have a leak.

     If you are sure none of the upstream flow system components are leaking, check for leaks at the column fitting and plugged inlet. If you find leaks, tighten the fittings and repeat the leak test.

Note If you can find no other leaks, the ECD itself is probably leaking. The ECD cannot be disassembled without special license from the Nuclear Regulatory Commission or Agreement State Licensing Agency (USA only).

Troubleshooting contamination problems

Persistent problems with high background or ghost peaks with temperature programming are almost always due to contamination from dirty samples, consumables, or the carrier/makeup gas systems.

Begin with the procedure in chapter 5, Volume 3, of the Operating Manual. If this procedure, which can be performed by the user, does not solve the problem, perform the following steps.

Ensure clean gas supplies

Before continuing, verify that the supply gases are of adequate purity.

1. Carrier and makeup purity must be >99.999%.
2. After confirming purity, verify that the tank regulators have stainless steel diaphragms (equivalent to Agilent part no. 8507-0407).
3. Install new 1/8-inch copper supply tubing—part no. 5180-4196. Many times "clean" tubing from other sources has caused high ECD background.

   At the same time, install new traps in both the carrier and makeup supplies. Place the moisture trap (part no. 5060-9084) closest to the tank and the indicating oxygen trap (part no. 3150-0528) closest to the GC. Leak test the entire plumbing setup very carefully.

Isolate problem to carrier or makeup gas supplies

Determine what components of the apparent contamination are from the carrier vs. makeup systems in the GC. Sharp, well-resolved peaks that elute during a temperature program *with no injection* are from the carrier/inlet system. Broader "humps" in the baseline are usually from the makeup system. Overall high background (>500 Hz) can be contaminated gas from either the carrier or makeup supply or a contaminated detector cell.

Remove the column from the detector and inspect the installation of the makeup gas adapter. It is quite common for the adapter to be installed too low. To check this, measure from the bottom of the 1/4-inch Swagelok nut to

the bottom of the hex of the makeup gas adapter. The measurement should be 19 to 20 mm. If it exceeds 22 mm, the adapter is installed incorrectly.

A ridge inside the cell can prevent the adapter from easily seating all the way. Wiggle the adapter while installing to allow it to go all the way in. Always check the nut to hex measurement to be sure.

Evaluate the makeup side

1. Remove the makeup adapter from the detector. Unscrew the tip and remove the Gigabore liner. Inspect the adapter body for carbon (graphite) deposits. Remove all graphite deposits and clean the adapter thoroughly with methanol. Soak the removable tip in methanol.
2. Reassemble the adapter *without* the gigabore liner. Install a 5-inch length of narrow bore column, capped with a new septum, so that the other end extends 1 mm past the tip of the adapter. Use a Vespel column ferrule rather than graphite. Tighten the tip just past finger tight with *clean* pliers. Clean the whole assembly with methanol before installing.
3. Install the capped-off makeup adapter, using a new 1/4-inch Vespel ferrule. Be sure it is fully seated—check the measurement. Retighten after the detector has been heated.
4. Set the makeup flow to the original setpoint and bake out the detector at 350°C for 1 hour. During this time, put the inlet in split mode with 200 to 300 mL/min split vent flow (gas saver off) and bakeout the inlet at 275°C. Bakeout the column at its appropriate temperature.
5. When bakeout is done, *do not* reconnect the column to the detector. Make a series of blank runs with the user's method. If the baseline is acceptable—free of peaks and humps and under 1000 Hz throughout a temperature program—then the detector and makeup system are clean.

   Any unacceptable baseline problems could indicate contaminated makeup gas, EPC module, makeup adapter, or detector. These must be addressed before continuing.

If the 6890 GC was manufactured before 6/97, the EPC modules may have O-ring contamination. See Service Note G1530-14 and follow the procedure given. Replace the EPC module.

Evaluate the carrier side

After the detector and the makeup system have been determined to be clean, evaluate the carrier, inlet, and column.

1. Remove the makeup adapter and capped-off column from the detector. Discard the "cap". Place a new ferrule on the column, trim the column end, and install it so that 1 to 2 mm extends past the adapter tip.

   It is best not to use the gigabore liner. A better solution is the mixing liner (part no. G2397-20540) for the μ-ECD. Use the column installation instructions for the μ-ECD in Volume 1 of the Operating Manual.

2. Wipe off the entire makeup adapter with methanol. Install it fully—check the measurement to be sure.

3. Bake out the entire system for another hour at these conditions:

   - Detector 350°C
   - Inlet Split mode, 275°C
   - Column An appropriate temperature

4. Reload the user's method and make a series of blank (no injection) runs to see if the problem has been cured. Note that a single, well-resolved peak could be due to the O-ring contamination problem, mentioned earlier. Address per service note.

5. If the contamination persists, perform a complete inlet maintenance, including thorough cleaning of the shell weldment. Replace the gold seal and liner. Install a known good 30 m/320 μm HP5 checkout column to rule out column contamination.

6. Peaks from the inlet side are usually due to contaminated carrier gas supply, EPC module, insert weldment, inlet or liner, or column.

Maintaining an ECD detector

ECD bakeout

If your ECD baseline is noisy or the display frequency is too high (i.e., ≥100), you should perform a thermal cleaning (also called a "bakeout") of the detector. Before performing a bakeout, verify that the carrier supply gas and flow system are leak- and contaminant-free.

Caution 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 these other procedures, causing possible hazardous exposure to β- and x-radiation (bremsstrahlung).

WARNING To prevent possible hazardous contamination of the area with radioactive material, the detector exhaust vent must always 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.

1. Record the ECD "Output" value from the GC display. If the number is equal to or greater than 100, continue with this procedure.
2. Turn off the anode purge and makeup gas flows.
3. Remove the column from the detector.
4. Cap the bottom of the makeup gas adapter with a blank column ferrule and column nut.
5. Set the makeup gas flow rate between 50 and 60 mL/min. Set the detector temperature between 350 and 375°C.

6. Set the oven temperature to 250°C.
7. Allow thermal cleaning to continue for several hours, and then cool the system to normal operating temperatures.

Performing a radioactivity leak test (wipe test)

ECDs must be tested for radioactive leakage at least every six 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 (part no. 18713-60050) is supplied with each new ECD. Refer to the information card supplied in the Wipe Test Kit for instructions on performing the wipe test.