25 The Micro-Cell Electron Capture Detector

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Regulatory and Safety Information

This chapter describes the micro-cell detector (μ-ECD).

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

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

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

The ^{63}Ni isotope

The radioactive isotope used in the cell is ^{63}Ni. It is plated onto the inner surface of the cell body and is solid at temperatures used in chromatography. Some other properties are listed in Table 67.

Table 67. Properties of ^{63}Ni

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

ECD licenses

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

Specific License

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

General License

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

Certain restrictions apply to General Licenses:

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

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

µ-ECD warnings

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

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

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

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

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

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

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

Safety precautions when handling μ-ECDs

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

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

General Information

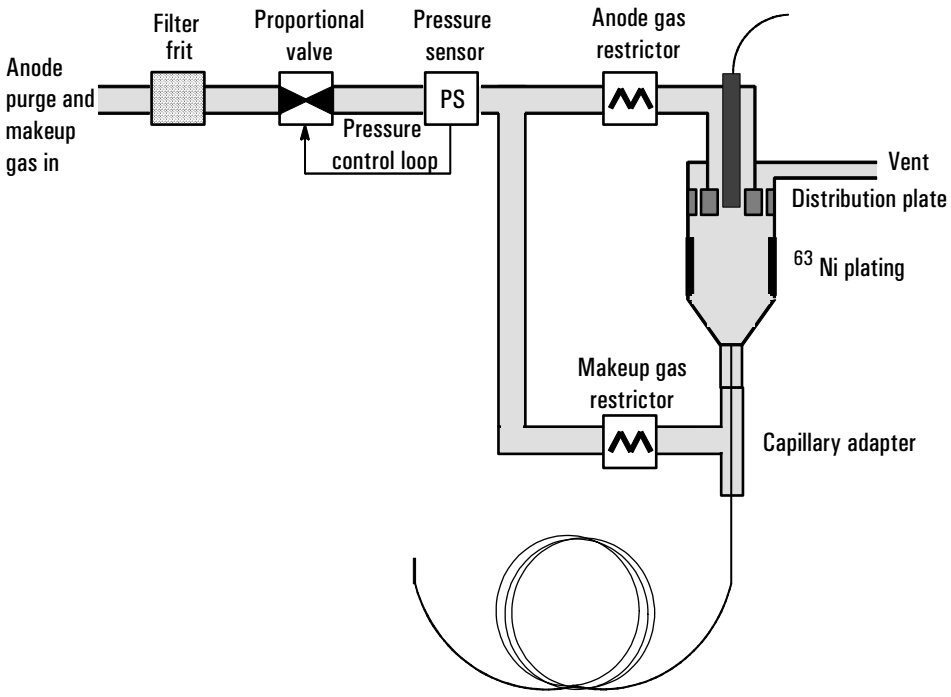


Figure 87. μ-ECD pneumatics

Linearity

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

Detector gas

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

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

Temperature

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

Electrometer

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

Operating the μ-ECD

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

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

Use the information in Table 68 when selecting temperatures and flows. Maximum source pressure must not exceed 100 psi. Use the maximum source pressure to achieve maximum makeup flow rate.

Table 68. Operating Parameters

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

Notes

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

Procedure: Operating the μ-ECD

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

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

Caution Detector electronics depend on the correct gas configuration.

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

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

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

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

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

   If your capillary column is *not defined*, only constant makeup flow is available. Enter a makeup gas flow.

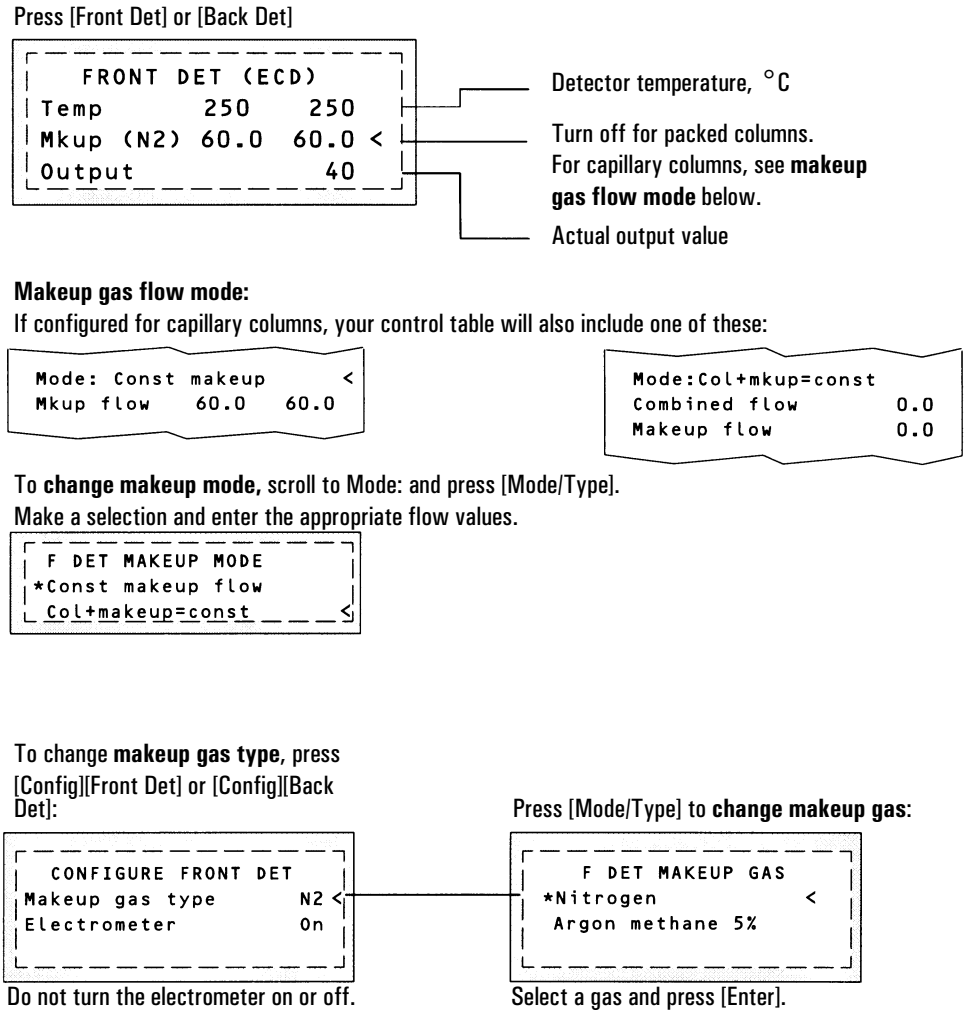


Figure 88. μ-ECD control table

Checkout Conditions and Chromatogram

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

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

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

µ-ECD checkout conditions

Column and sample

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

Inlet

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

Split/Splitless

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

Inlet, continued

PTV

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

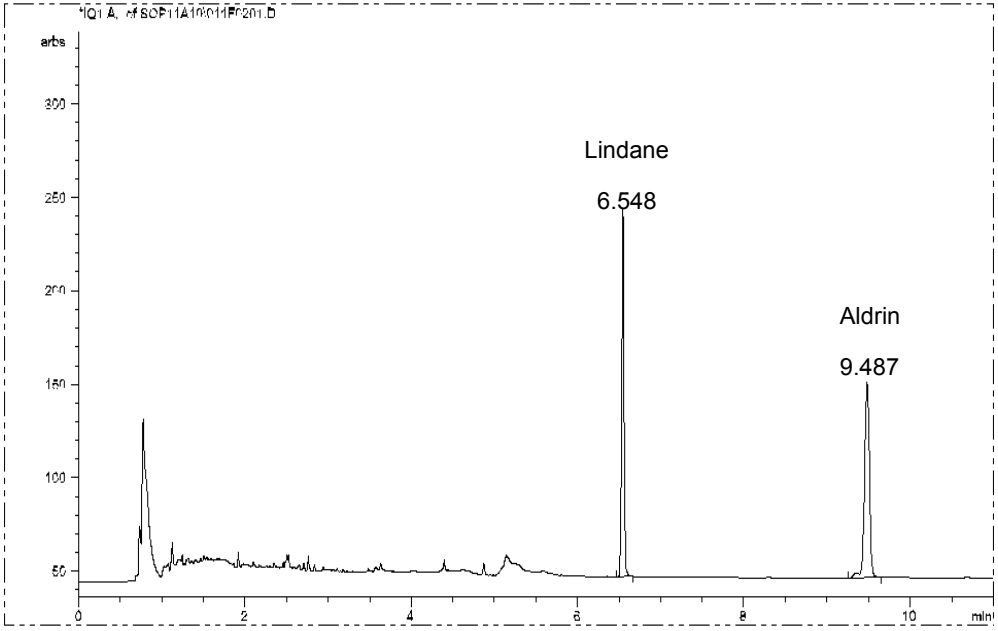
Detector

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

Oven

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

Typical µ-ECD checkout chromatogram



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

Maintaining the Detector

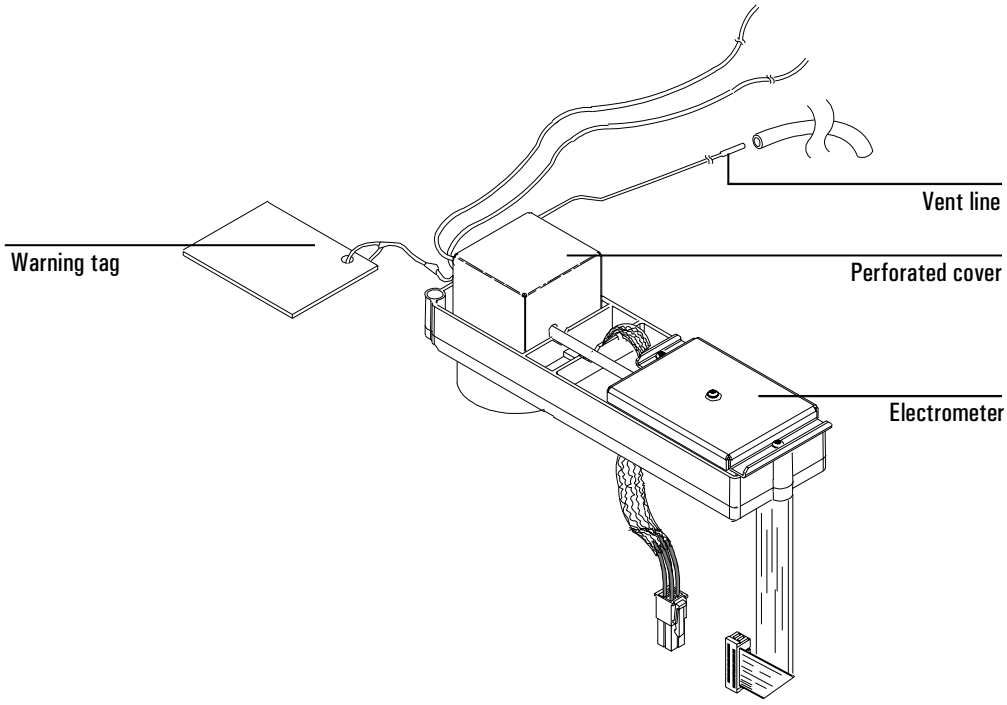


Figure 89. The μ-ECD

Correcting performance problems

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

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

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

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

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

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

3. The detector itself is not a likely source of leaks, so you should leak test the inlet if the output reading is still abnormal. See the maintenance material for your inlet in "The Split/Splitless Inlet", "The Purged Packed Inlet", "The Cool On-Column Inlet", "The Programmable Temperature Vaporization Inlet", "The Volatiles Interface".

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

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

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

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

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

5. Check for contamination:

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

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

   c. If the output is abnormal, then the detector is contaminated. Perform a thermal bake out to decontaminate the detector. The procedure is described later in "Thermal cleaning".

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

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

Checking for gas leaks

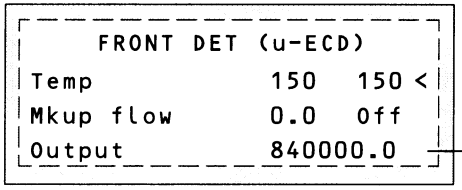
The detector is an unlikely leak source. If you suspect that there is a leak in your GC system and have checked the gas plumbing to the GC, the inlet, and the column inlet and detector connections without finding it, follow this procedure to test the detector.

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

Materials needed:

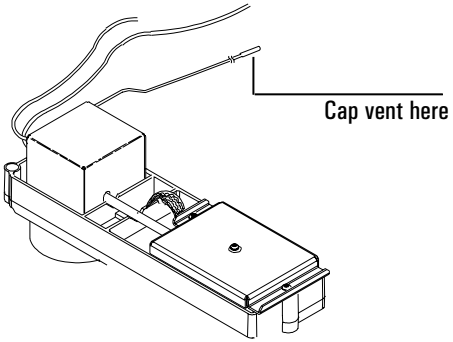
- A vent plug (part no. 5060-9055)
- An electronic leak detector capable of detecting your carrier gas

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



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

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



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

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

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

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

Thermal cleaning

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

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

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

Materials needed:

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

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

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

Performing a wipe test (radioactivity leak test)

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

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