

## **Electron Capture Detector Troubleshooting Tips**

**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  $^{63}\text{Ni}$  may be removed during these other procedures, causing possible hazardous exposure to radiation.

**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.

### **TROUBLESHOOTING:**

Performance problems associated with the Electron Capture Detector include (but are not limited to) loss of sensitivity (real, or perceived), high signal background, noisy baseline, and chromatographic peaks or humps that are not characteristic of the samples being introduced in the inlet. If the problems are not accompanied by an increase in the signal output, as read from the front keypad of the GC, then suspect the detector only after the other parts of the chromatographic system have been checked.

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| <p>This document is believed to be accurate and up-to-date. However, Agilent Technologies, Inc. cannot assume responsibility for the use of this material.<br/>The information contained herein is intended for use by informed individuals who can and must determine its fitness for their purpose.</p> |
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Before starting extensive troubleshooting, consider first the nature of the problem:

1. If recent changes were made in the system such as changing carrier or detector gases, performing inlet or column maintenance, or changing the column, investigate the possibilities that contamination or leaks were introduced.
2. If the problem has been chronic and is now acute enough to interfere with analysis, suspect contamination, column degradation, or ultimately a bad ECD Cell.

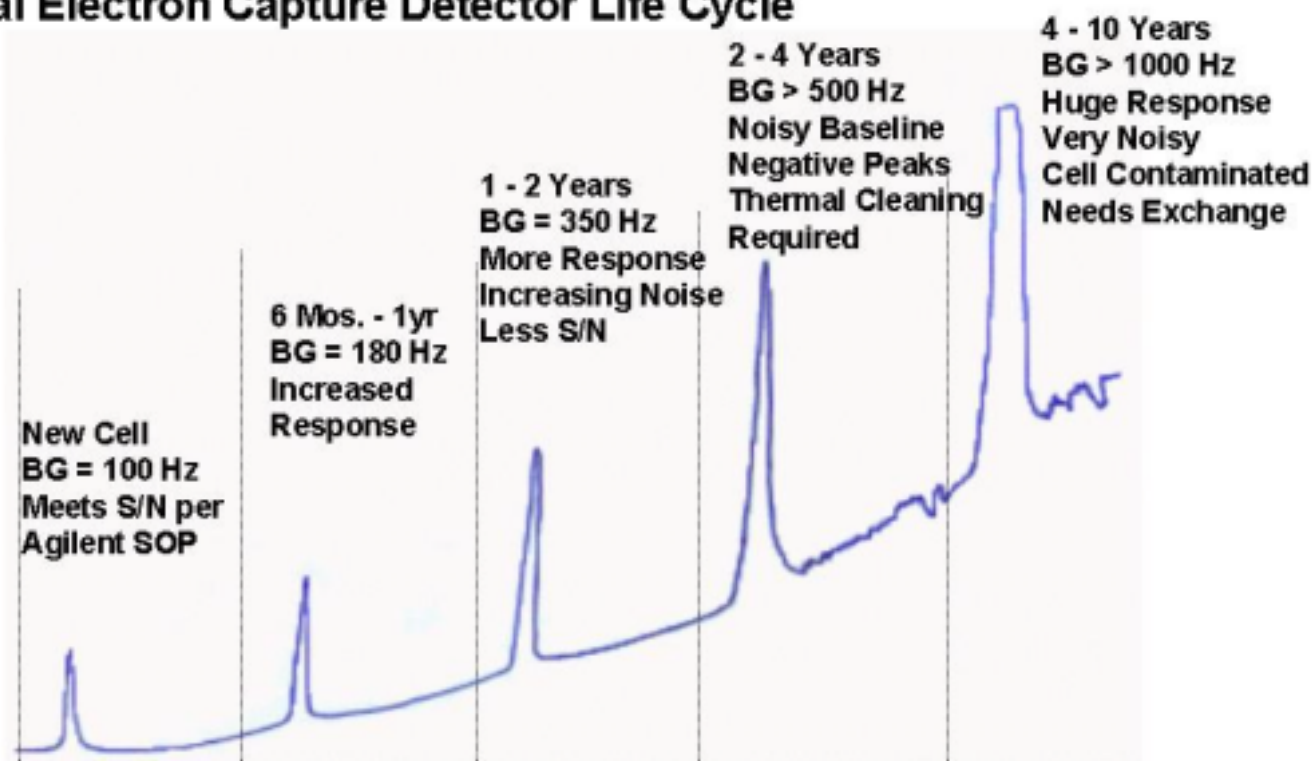
### DISPLAY SIGNAL EVALUATION:

The first consideration is the signal value when the GC is in the idle state. The “idle” signal level is a function of the type and quality of the carrier and detector gases, as well as the flows and application. It may be different for different situations but as a general rule, the following values are presented as a guideline.

Signal viewed from the GC display, at ambient oven temperature:

| <u>5890</u> | <u>6890 ECD</u> | <u>6890 u-ECD</u> |                                                                                                                                                              |
|-------------|-----------------|-------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <10         | <20             | <200              | ECD is likely in a good state of health.                                                                                                                     |
| 10-40       | 20-80           | 200-400           | Slightly elevated, no cause for concern at this point. Signal still in “good” range.                                                                         |
| 40-80       | 80-150          | 400-1000          | System showing signs of contamination from gases, column, or samples. If the signal increases in response to increased oven temperature, suspect the column. |
| 80-200      | 150-300         | 1000-2000         | Suspect more severe contamination, follow the troubleshooting guidelines.                                                                                    |
| >200        | >300            | >2000             | If the following procedures do not work, suspect the ECD Cell.                                                                                               |

## Typical Electron Capture Detector Life Cycle



Note: The above example is targeting the 6890 micro-ECD.

### SENSITIVITY

If the ECD is in the “good” signal range, and sensitivity is the issue, the problems quite likely may be in the injection port or column. The following issues may need to be considered:

1. If using a split/splitless injection port verify that the mode has not changed and that the valve is functioning. This can be accomplished by verifying that there is a corresponding change in the response by making split and then splitless injection, all other method parameters remaining the same. If using split mode, verify the split flow using a flow meter.
2. Confirm that complete injection port maintenance has been performed. This includes trimming and re-installing the column.

3. Verify that the injection syringe is not clogged. If the method is one using an external sampler (headspace, purge and trap, valve, etc.) make a direct injection of concentration similar to that introduced by the sampler. Rule out a problem with the sampler.
4. Inspect the mixing liner in the 6890 makeup gas adapter – a small piece of graphite or sample contamination will reduce sensitivity.

### CONTAMINATION:

If the problem includes stray or ghost peaks, broad humps that do not resemble chromatographic peaks, or elevated signal, as determined from the guidelines above, contamination may be an issue. Resolved ghost peaks are usually from the inlet/column while broad humps are usually from the makeup gas system.

1. Verify that gas supplies are clean. Carrier and makeup gas are recommended to be >99.9995% purity. Even ultra high purity gases should have traps. Moisture, oxygen, hydrocarbon traps are recommended. Check traps for plastic composition or O-Ring seals – these can introduce contamination.
2. Run a series of blank runs:  
  
Solvent only – If symptoms persist, replace the syringe. If symptoms are gone, check the sample preparation procedure and solvents.  
  
No injection (Remove Syringe from ALS) – If symptoms persist, the problem is in the GC, column or carrier gas. Proceed to step 3. If symptoms are gone, the problem is in the sample/solvent or the solvent is causing the contamination in the injection port to be released at injection.
3. Remove the column from the detector and cap the base of the makeup gas adapter using no-hole ferrule (p/n 5181-7458). If this is a packed column application, use a column with no packing. Verify that the makeup gas flow is present for capillary column applications or column flow for packed column applications.
4. If the signal changes significantly by simply turning the oven (fan) on and off, suspect a crack in the makeup gas line. STOP HERE and evaluate replacing the makeup gas adapter. Baking the detector with a leak can cause damage to the ECD Cell.

5. Run the GC temperature program. The baseline should be free from peaks or humps – if not, suspect the makeup gas if a capillary system or carrier gas if packed system. The makeup weldment or EPC module could be contaminated – proceed with bakeout.
6. Bake the detector at 350C for 1 hour, taking note of the initial and final signal level during this period. It is timely to increase the oven and injection port temperatures and bake the (capillary) column with flow simultaneously. If the ECD signal decreases immediately after capping the detector, suspect contamination prior to the ECD. If not, but the signal decreases during the bake period, consider contamination in the makeup plumbing or detector. If the signal decreases significantly during the bake, it may be advisable to extend the bake period until the signal stabilizes.
7. If the signal does not decrease during the bake, the possibilities include a leak in the detector or makeup system, or a bad ECD Cell. (Remember that a leak in the makeup area will be an inward or aspirating leak and may not be detected by normal means.)
8. After bake, before reinstalling the column, perform a few blank runs with a typical method. If the baseline is acceptable, the detector and makeup system may be deemed clean. If not, further evaluation of makeup gas quality, leaks in the makeup gas plumbing, and ECD Cell itself are in order.

These suggestions are intended to be used as guidelines for troubleshooting hardware related problems with Electron Capture Detectors. If the problem is not resolved using these techniques, [Call Agilent Technical Support](#) for further assistance or on-site service. Please note that Agilent Engineers are not permitted to transport ECD Cells. These arrangements must be made with Bench Repair.