



## Troubleshooting Contamination problems with an Electron Capture Detector

### 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.

#### 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 is it from 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.

#### How to Install Make up Gas Adapter.

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. Please refer the figure.

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## Evaluate the makeup side

1. Remove the column from the makeup adapter and remove the makeup adapter from the detector. Unscrew the tip and remove the Gigabore liner. It is best not to use the gigabore liner. A better solution is the mixing liner (part no. G2397-20540) for the  $\mu$ -ECD. 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. Tighten the tip just past finger tight with *clean* pliers. Clean the whole assembly with methanol before installing.
2. Reconnect the makeup gas adapter using new 1/4-inch Vespel ferrule. Be sure it is fully seated— check the measurement. Retighten after the detector has been heated. Instead of the column blank the bottom of the makeup gas adapter with blanking nut (P/N 5181-8830) and Vespel/Graphite ferrule (P/N 5181-3308)
3. Set the makeup flow to the original set point 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 bake out the inlet at 275° C. Bake out the column at its appropriate temperature.
4. When bake out 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 your 6890 GC was manufactured before 6/97, the EPC modules may have O-ring contamination. Please check with Agilent representative and 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 blanking nut and ferrule from the make up gas adapter and reconnect the column properly with new ferrule. You can trim the column end and wipe it with moist tissue paper moist with methanol.
2. Bake out the entire system for another hour at these conditions:
  - Detector 350° C
  - Inlet Split mode, 275° C
  - Column An appropriate temperature
3. 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.
5. If the contamination persists, perform complete inlet maintenance, including thorough cleaning of the shell weldment. Replace the gold seal and liner. Install a known good 30 m/320  $\mu$ m 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.  
Replace the carrier gas cylinder with new cylinder and check.

