Thermal Conductivity Detector

Troubleshooting Tips

The thermal conductivity detector (TCD) detects the difference in thermal conductivity between column effluent flow (carrier gas + sample components) and a reference flow of carrier gas alone; it produces a voltage proportional to this difference. The voltage then becomes the output signal to the connected chart recording or integrating device.

The two gas streams are switched over the filament at a rate of about 5 times per second, by the switching solenoid valve. An audible “clicking” can be heard when the detector is on.

Crucial to the proper response of the TCD is:

- Temperature of the detector
- Flow rate ratio of the column + makeup gas flow and the reference flow
- Filament resistance

If either of these factors is not optimal, then the response of the TCD will be compromised.

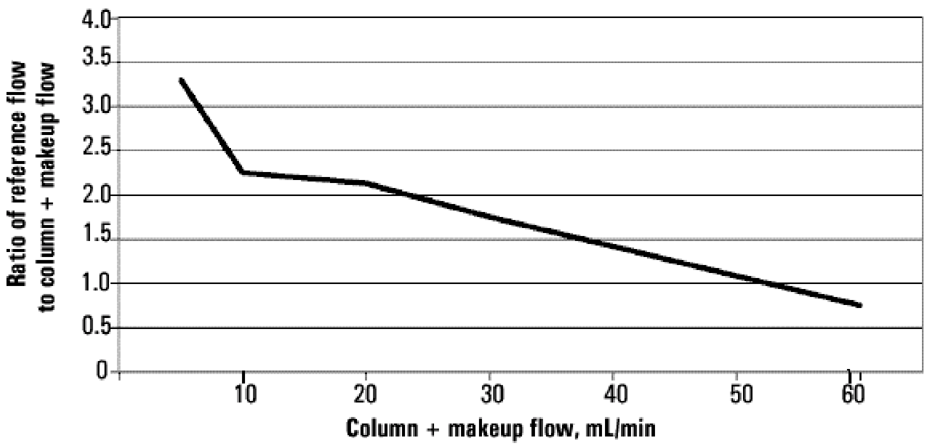
<u>Temperature</u>

The TCD should be operated at the lowest possible temperature (limited by highest boiling components condensing inside the detector). This temperature will yield maximum sensitivity and lead to increased filament lifetime. For the 6890/6850 the TCD will not turn on below a temperature of 150 degrees C.

Flow rate ratios

Reference gas and makeup gas flow must be the same as the carrier gas, and for the 6890/6850 the gas type must be specified in the inlet and detector control tables.

- The chart below is a good reference for selecting flow rates for the 6890/6850 using a capillary column.

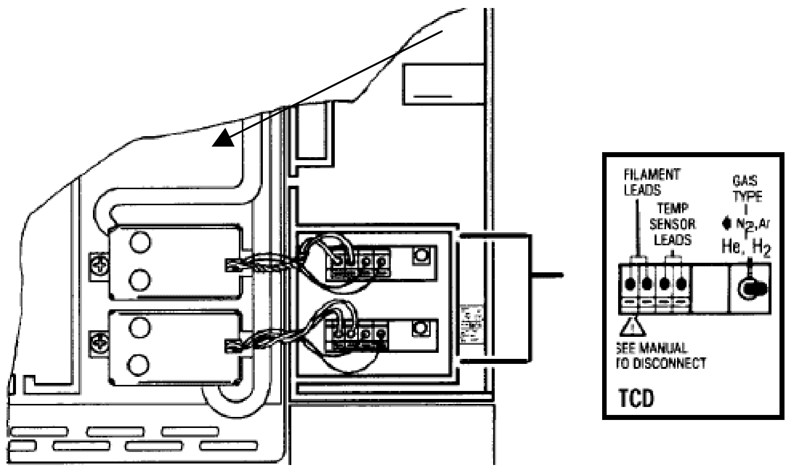


- For a 5890 GC using a capillary column, the reference flow should be 3 times the total flow of the column + makeup gas flow. Therefore, if column + makeup flow is 5 ml/min then the reference flow should be set at 15 ml/min.
- For a 5890 and 6890 GC using packed columns, the reference flow should be calculated as 1.5 times the total flow of the column flow. Therefore, if the column flow is 30 ml/min then the reference flow should be 45 ml/min.

Filament Resistance

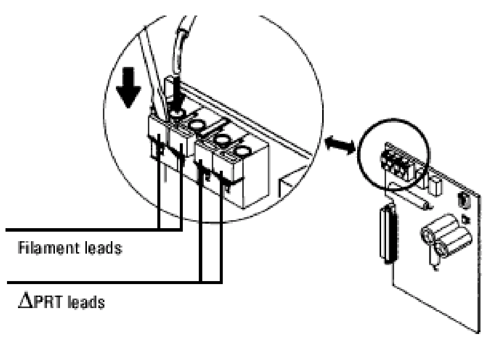
The resistance of the TCD filament should read typically around 10 ohms (cold) and 11-13 ohms (hot). A filament that is broken or “open” would read infinite resistance. Chemically active compounds such as acids and halogenated compounds should be avoided when using the TCD since they attack the filament, change the resistance and thus, permanently change the detector sensitivity.

<u>5890 filaments leads</u>



Special care should be taken when disconnecting the TCD filament leads, which run from the detector to the detector interface card. Use a small blade screwdriver to push down on the connector tabs while pulling out the wires.

<u>6890/6850 filament leads</u>



NOTE: THIS SHOULD BE PERFORMED BY TRAINED INDIVIDUALS, IF YOU ARE UNSURE OF HOW TO TAKE THESE MEASUREMENTS THEN STOP HERE AND CALL AGILENT!

The TCD filament can be permanently damaged if gas flow through the detector cell is interrupted while the filament is operating. The detector should always be off whenever changes/adjustments are made that affect gas flows through the detector. Likewise, a "leak free" environment should be maintained since, oxygen can also permanently damage the filament as well.

Troubleshooting TCD Performance problems

Temperature, Flow ratio and Filament resistance are the first things to examine when troubleshooting performance problems with the TCD. There are many other factors that affect performance within the GC system. Problems such as loss of sensitivity, high/noisy signal background and the presence or absence of peaks can be attributed to sample introduction, column and electronic problems. A systematic troubleshooting plan should be implemented prior to immediately assuming that the detector cell is at fault.

Sensitivity

Factors affecting sensitivity of the detector not previously mentioned before.

1. For a 5890 GC, verify that the sensitivity setting is correct for the application being used.
2. Poor sensitivity can be caused be contamination of the GC flow system. Gases with 99.999 percent purity should be used. If an elevation of signal is observed with the GC in the idle state and at ambient temperature then contamination or a leak in the system should be investigated. A thermal bake out of the detector cell is the first step in resolving an elevated signal after it is certain that the gases are not suspect and the system is leak free.

Noise/Spikes

Baseline noise, chromatographic upsets and spiking can result from the following:

1. A valve time table. (If the GC is equipped with gas or liquid sampling valves.)
2. A malfunctioning or leaking solenoid valve. (The switching valve for the detector).
3. In some isolated cases, it has been found that "walkie-talkie" radios have been known to causing spiking in the baseline.
4. Power problems, poorly grounded power receptacles or power panels.
5. On a 5890, the carrier gas, He/N2, switch is in the wrong position.
6. Electronic problems due to poor connections, or faulty detector board.

Zero Offset

The signal level of the detector when it is in the “idle” state (detector at proper operating temperature and the GC oven at ambient) is the reference point for troubleshooting the detector offset. If the detector is on, the solenoid is clicking and the offset is zero, then as previously mentioned, the ratio of the column + makeup gas flow to reference flow should be checked first. The filament continuity should then be checked next.

This is a basic troubleshooting guideline and in no way covers every problem that can be encountered when using the TCD. If problems persist after addressing these points then Agilent Service should be contacted.