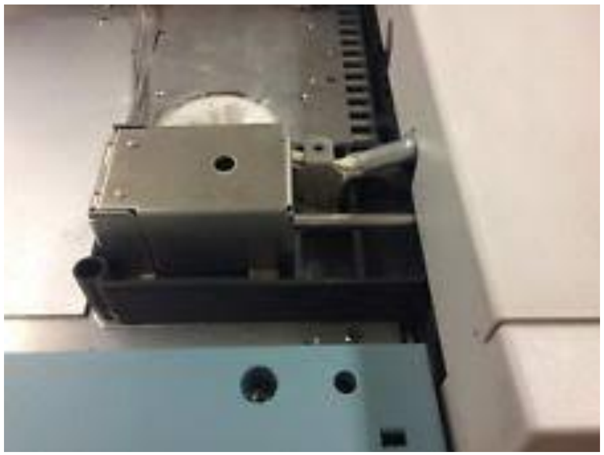
TCD (Thermal conductivity detector)



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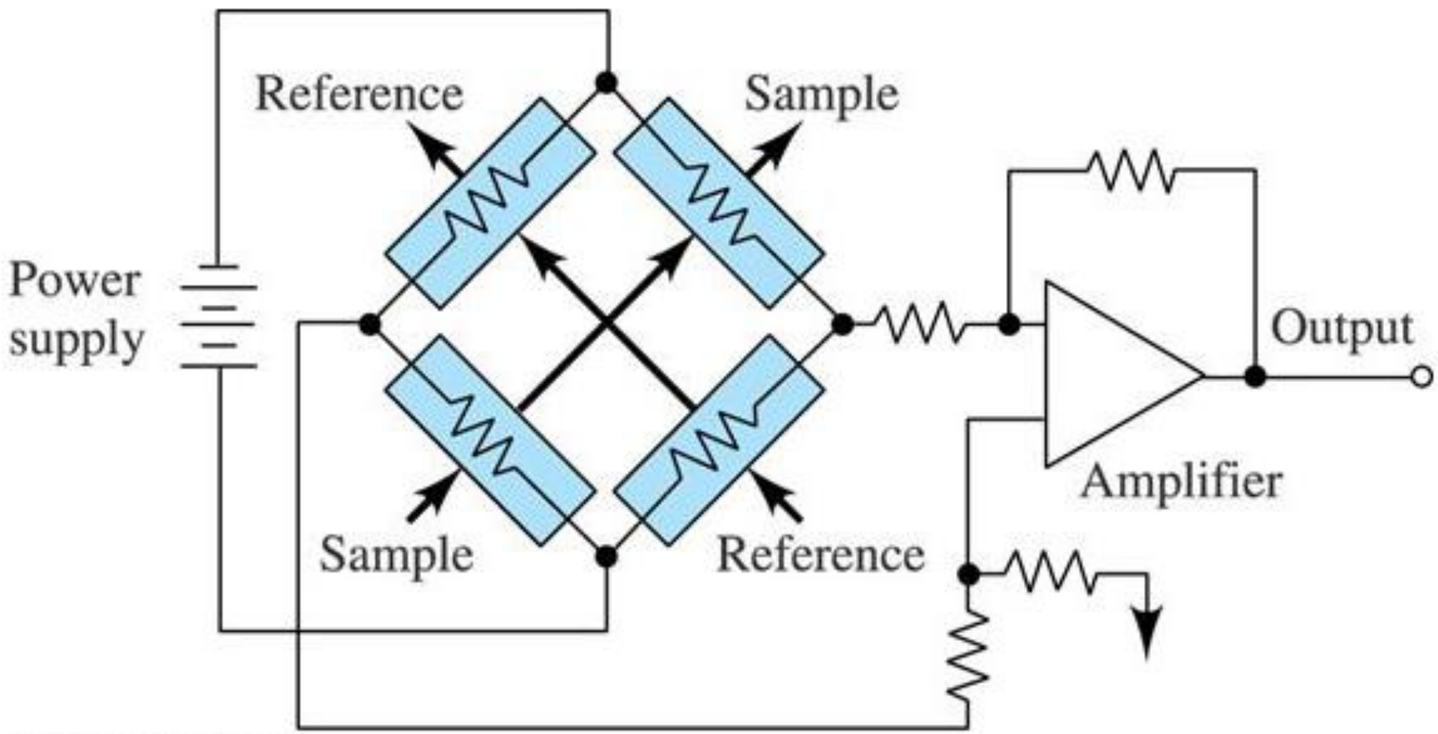
Principle:

The TCD compares the thermal conductivities of two gas flows—pure carrier gas (also called the reference gas) and carrier gas plus sample component.

This detector contains a filament that is heated electrically so that it is hotter than the detector body. The filament temperature is kept constant while alternate streams of reference gas and column effluent pass over it. When sample is added, the power required to keep the filament temperature constant changes. The two gas streams are switched over the filament and the power differences were measured and recorded.

When helium (or hydrogen) is used as carrier gas, the sample causes the thermal conductivity to fall. If nitrogen is used, the thermal conductivity usually goes up because most things are more conductive than nitrogen.

Schematic:


Reference
Sample
Power
supply
Output
Amplifier
Sample
Reference

Advantages:

Responds to all compounds

Adequate sensitivity for many compounds

Good linear range of signal

Simple construction

Signal quite stable provided carrier gas glow rate, block temperature, and filament power are controlled

Nondestructive detection

Conditions that prevent the detector from operating

Temperature set below 150°C

Broken or shorted filament

Reference gas flow set to 0

Filament passivation

The tungsten-rhenium TCD filament has been chemically passivated to protect against oxygen damage. However, chemically active compounds such as acids and halogenated compounds may attack the filament. The immediate symptom is a permanent change in detector sensitivity due to a change in filament resistance.

Such compounds should be avoided. If this is not possible, the filament may have to be replaced frequently.

Negative polarity

Sample components with higher thermal conductivities than the carrier gas produce negative peaks. For example, helium or hydrogen form a negative peak with nitrogen or argon-methane as the carrier gas.

Analyzing for hydrogen

Hydrogen is the only element with thermal conductivity greater than helium, and mixtures of small amounts of hydrogen (<20%) in helium at moderate temperatures exhibit thermal conductivities less than either component alone.
Analyzing for hydrogen with helium carrier gas, a hydrogen peak may appear as positive, negative, or as a split peak.

There are two solutions to this problem:

- Use nitrogen or argon-methane as carrier gas. This eliminates problems inherent with using helium as carrier, but causes reduced sensitivity to components other than hydrogen.
- Operate the detector at higher temperatures—from 200°C to 300°C.

Parameters

Gas type Flow range
Carrier gas
(hydrogen, helium, nitrogen)
Packed, 10 to 60 mL/min
Capillary, 1 to 5 mL/min
Reference
(same gas type as carrier)
15 to 60 mL/min
Capillary makeup
(same gas type as carrier)
5 to 15 mL/min—capillary columns
2 to 3 mL/min—packed columns

Detector temperature

<150° C, cannot turn on filament
Detector temperature should be 30°C to 50°C greater than highest oven ramp temp

Correcting TCD performance problems

If the TCD is displaying problems such as a wandering baseline, increased noise level, or changes in response on a checkout chromatogram, it is probably contaminated with deposits from such things as column bleed or dirty samples. The TCD is cleaned by a process known as bakeout. Bakeout should be performed only after you have confirmed that the carriergas and the flow system components are leak and contaminant free.

Caution Baking out the detector with a large air leak present can destroy the filament.

Procedure: Thermal cleaning

The only common maintenance procedure to perform on the TCD is thermal cleaning.

Caution Turn off the TCD and cap the detector column fitting to prevent irreparable damage to the filament caused by oxygen entering the detector.

1. Turn the detector off.
2. Remove the column from the detector and cap the detector column fitting.
3. Set the reference gas flow rate between 20 and 30 mL/min. Set the detector temperature to 400°C
4. Thermal cleaning to continue for several hours. Then cool the system to normal operating temperatures.