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The Thermal Conductivity Detector

General Information

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

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 five times per second and the power differences are 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.

Because the TCD does not destroy the sample during the detection process, this detector can be hooked up in series to a flame ionization detector or other detector.

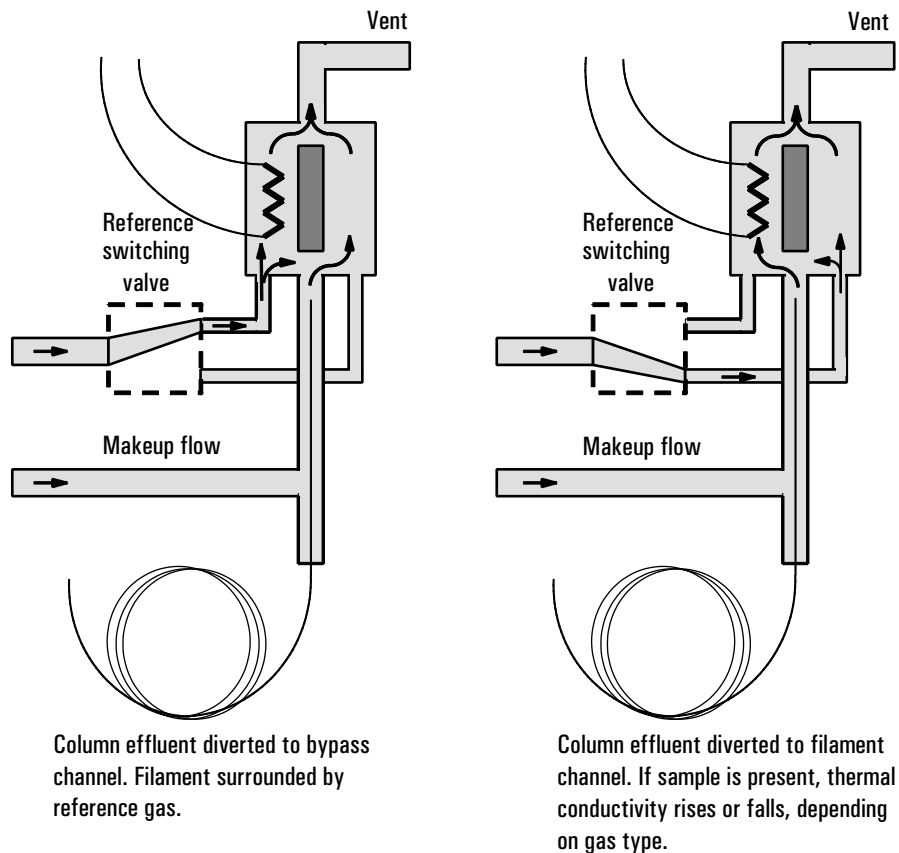


Figure 76. TCD — Conceptual diagram

TCD pneumatics

[Figure 77](#) shows the pneumatics design of the TCD.

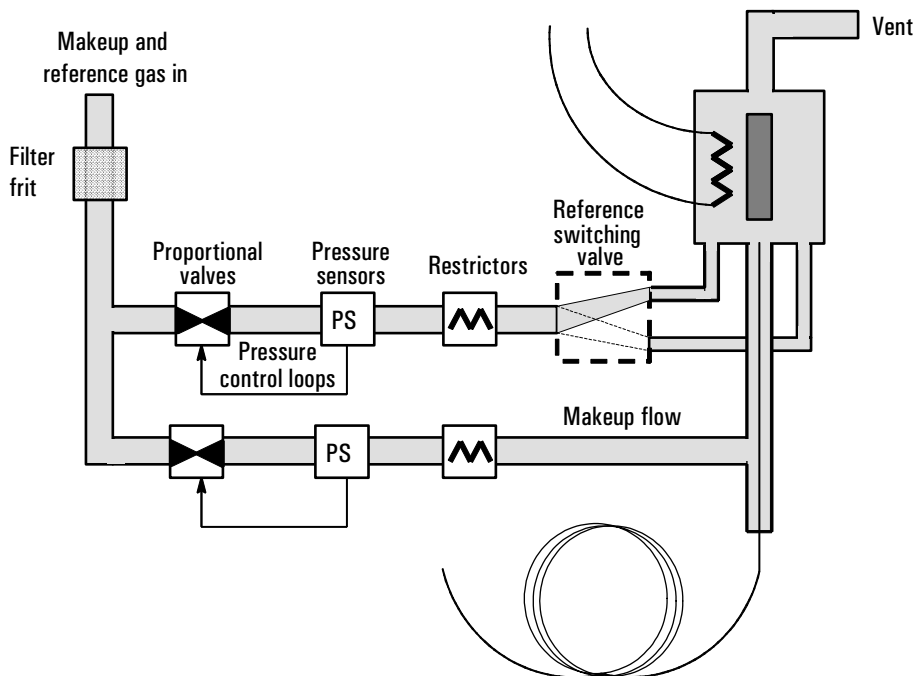


Figure 77. TCD pneumatics

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.

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

Carrier, reference, and makeup gas

Reference and makeup gas must be the same as the carrier gas, and the gas type must be specified in both the inlet and detector control tables.

When using packed columns, we recommend a small makeup gas flow (2 to 3 mL/min) to get the best peak shapes.

Use [Figure 78](#) to select a value for reference gas flow for either capillary or packed columns. Any ratio within ± 0.25 of that in the figure is suitable.

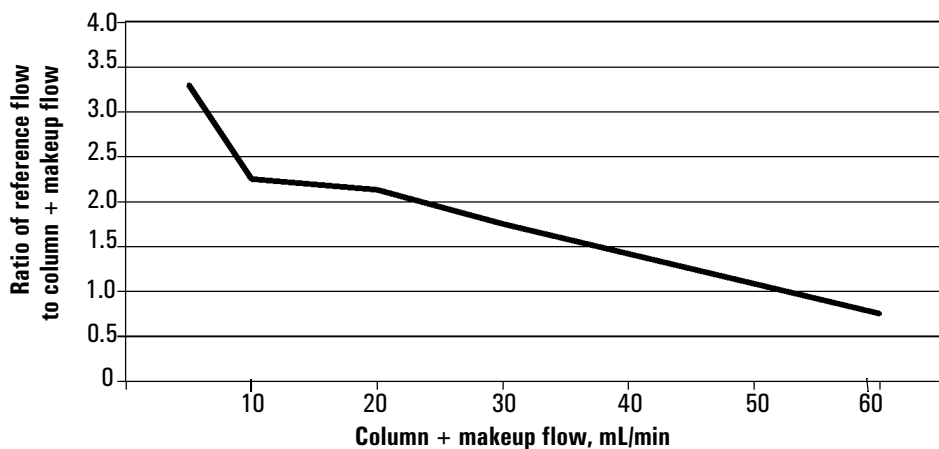


Figure 78. Selecting the reference gas flow

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.

Neg polarity ON inverts the peak so the integrator or ChemStation can measure it. Neg polarity can be a run table entry; see ["Run time programming"](#).

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. If you are 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.

You can find the correct detector operating temperature by analyzing a known range of hydrogen concentrations, increasing the operating temperature until the hydrogen peak exhibits normal shape and is always in the same direction (negative relative to normal response to air or propane) regardless of concentration. This temperature also ensures high sensitivity and linear dynamic range.

Because hydrogen peaks are negative, you must turn negative polarity on at appropriate times so the peak appears positive.

Operating the TCD

Use the information in [Table 62](#) when selecting temperatures and flows for the TCD. Use [Figure 79](#) to locate minimum source pressures. If you have an EPC detector, you must add 10 psi (69kPa) to the source pressure on the chart.

Table 62. Recommended Flow Rates and Temperatures

Gas type	Flow range
Carrier gas <i>(hydrogen, helium, nitrogen)</i>	Packed, 10 to 60 mL/min Capillary, 1 to 5 mL/min
Reference <i>(same gas type as carrier)</i>	15 to 60 mL/min See Figure 79 to select a value.
Capillary makeup <i>(same gas type as carrier)</i>	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 temperature.	

Gas pressures

Choose a flow, find a pressure, set source pressure 10 psi (70 kPa) higher.

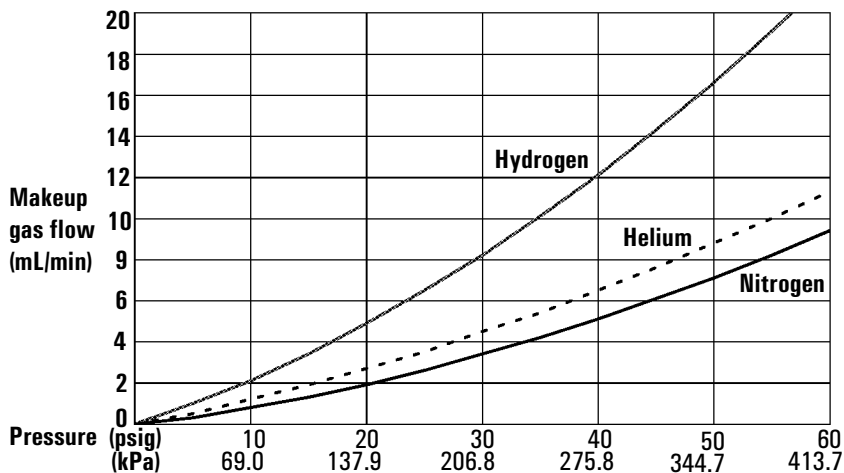
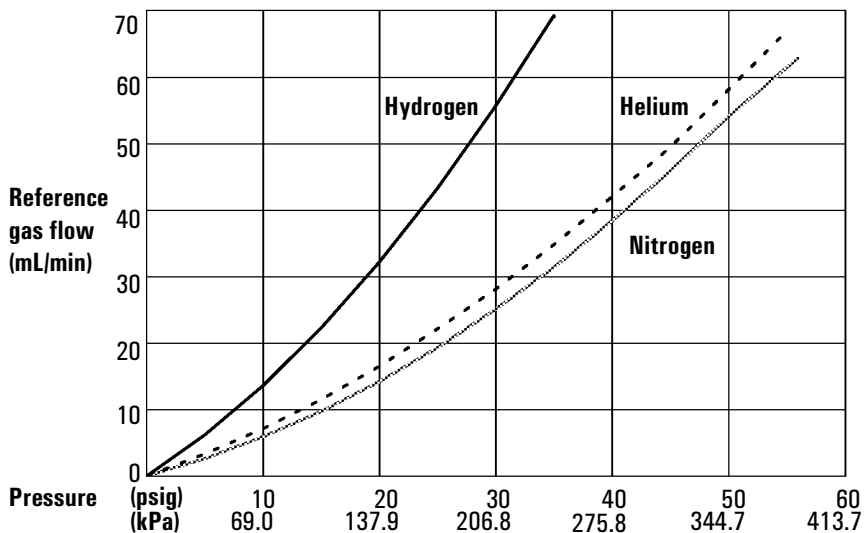


Figure 79. Typical pressure/flow relationships, reference and makeup gases (at 25°C and 1 atmosphere of pressure)

Operating the TCD

Press [Front Det] or [Back Det].

FRONT DET (TCD)		
Temp	24	Off
Ref flow	0.0	Off
Mkup (He)	0.0	Off
Filament		Off <
Output	0.0	
Neg polarity		Off

Temperature °C or off

Reference gas flow, mL/min or off
Turn off for packed columns.*
For capillary columns, see **makeup gas flow mode** below.

Press [On] or [Off].

Shows output value

Reverse polarity, turn on or off.
See "[Instrument Automation](#)".

Makeup gas flow mode:

If configured for capillary columns, your control table will also include one of these:

```
Mode: Const makeup <
Mkup flow 0.0 Off
```

```
Mode: Col+mkup=const
Combined flow 0.0
Makeup (He) 0.0
```

To **change makeup mode**, scroll to Mode: and press [Mode/Type].

Make a selection and enter the appropriate flow values.

```
F DET MAKEUP MODE
*Const makeup flow
Col+makeup=const <
```

To **view makeup/reference gas**, press

[Config][Front Det] or [Config][Back Det]:

```
CONFIGURE FRONT DET
Mkup/ref type He <
```

To **change makeup/reference gas**,

press [Mode/Type]:

```
F DET MAKEUP/REF GAS
Helium <
Hydrogen
Nitrogen
```

* A makeup flow of 2 to 3 mL/min improves peak shapes.

Select the appropriate gas.

Figure 80. TCD control table

Procedure: Using the TCD

This procedure assumes that detector support gases are connected, the system is leak-free, and a column is installed. Before operating the detector, set oven temperature, inlet temperature, and inlet/column flow.

1. Press [Front Det] or [Back Det] to open the detector control table.
2. Set the detector temperature. Do not set higher than the maximum temperature allowed for the column because part of the column passes through the heated block and into the cell.
3. Verify that gas type is the same as that plumbed to your instrument (next to Mkup line of control table). Change the gas type, if necessary (["Makeup gas flow"](#)).

Caution

Detector electronics depend on the correct gas type configuration.

4. Set the reference gas flow rate.
5. If you are using **packed columns**, turn off the makeup gas (or proceed to Step 6 and enter 2 to 3 mL/min, see ["Carrier, reference, and makeup gas"](#)) and proceed to Step 7.
6. If you are using **capillary columns**:
 - a. If your column is *defined* and connected to an EPC inlet, choose a new flow mode (["Makeup gas flow"](#)) if desired, and set the makeup gas flow or combined flow.
 - b. If your column is connected to a nonEPC inlet, enter a makeup gas flow. Only constant flow is available in this case.
7. Turn on the filament. Allow about 30 minutes for thermal stabilization. A longer period may be needed for the highest sensitivity.
8. If necessary, turn Negative polarity [On] to invert negative-going peaks. When a sample contains components giving both positive- and negative-going peaks, Neg polarity can be switched on and off during a run as a timetable event.

Short-cut procedure:
(assumes your setpoints are stored)

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

Checkout Conditions and Chromatogram

This section contains a typical examples 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.

TCD checkout conditions

Column and sample

Type	HP-5 30m \times 0.32mm \times 0.25 μm PN 19091J-413
Sample	FID Checkout 18710-60170
Injection volume	1 μL

Inlet

Temperature	250°C Purged/Packed or Split/Splitless Oven Track Cool On-Column 40°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	40°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 EPV inlets)
Purge time	0.75 min
Purge flow	60 mL/min

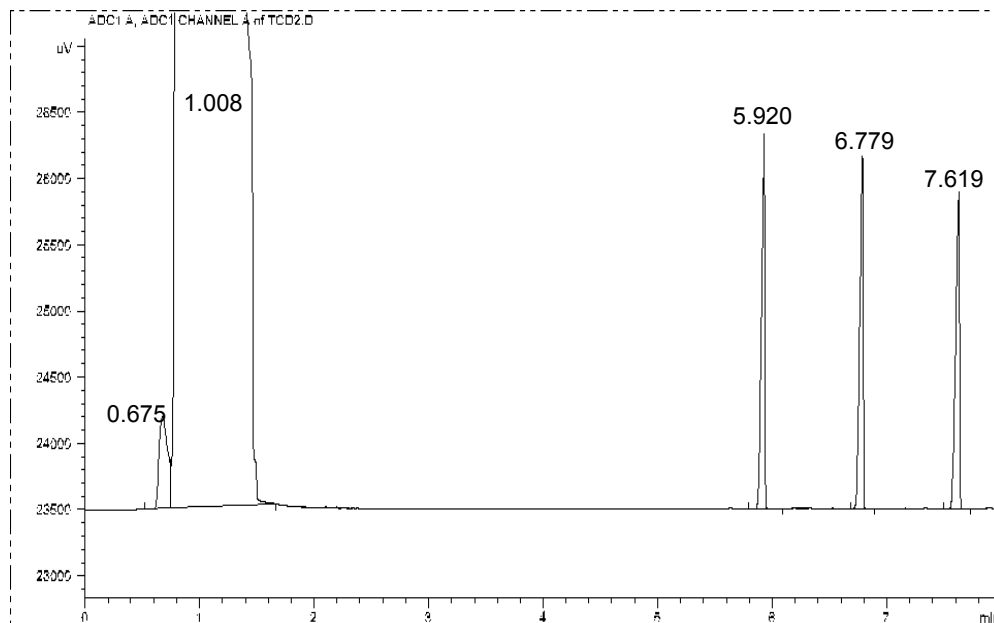
Detector

Temperature	300°C
Reference flow (He)	30 mL/min
Makeup flow (He)	2 mL/min
Offset	Should be < 30 display counts

Oven

Initial temp	40°C
Initial time	0 min
Rate 1	25°C/min
Final temp	90°C
Final time	0 min
Rate 2	15°C/min
Final temp	170°C
Final time	2 min

Typical TCD checkout chromatogram



Your retention times will differ, but peaks should resemble this example.

Maintaining a Thermal Conductivity Detector

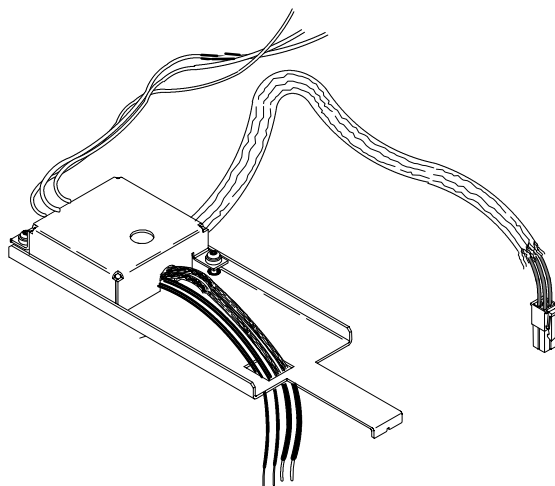


Figure 81. The TCD

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 carrier gas 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 you will need to perform on the TCD is thermal cleaning.

The TCD can become contaminated with deposits from such things as column bleed or dirty samples. A wandering baseline, increased noise level, or changes in response on a checkout chromatogram all indicate contamination.

Thermal cleaning is also known as **bakeout**. Bakeout should be performed only after you have confirmed that the carrier gas and the flow system components are leak and contaminant free.

Caution

You must 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.

FRONT DET (TCD)		
Temp	55	Off <
Ref flow	0.0	Off
Mode: Col + mkup = const		
Combined flow	0.0	Off
Makup flow		Off
Filament		Off
Output (off)		Off
Neg polarity		Off

4. Allow thermal cleaning to continue for several hours. Then cool the system to normal operating temperatures.