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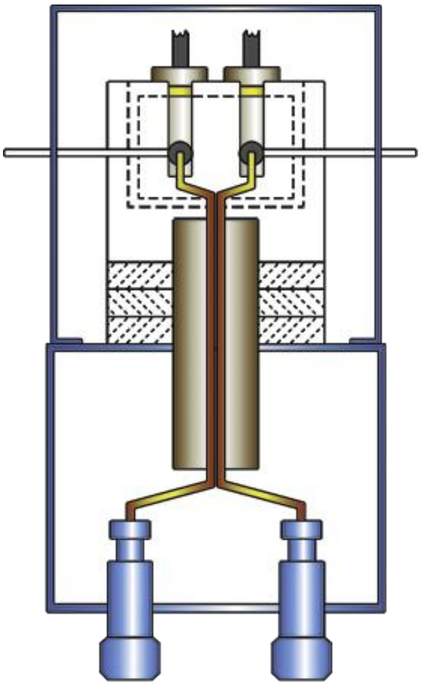
Детектор теплопроводности TCD



Thermal Conductivity Detector – TCD

Most of the early GC instruments were equipped with thermal conductivity detectors. They have remained popular, particularly for packed columns and inorganic analyses like H2O, CO, CO2, H2 and all inert gases.

Most of the early GC instruments were equipped with thermal conductivity detectors. They have remained popular, particularly for packed columns and inorganic analytes like H2O, CO, CO2, H2 and all inert gases. The TCD is a differential detector that measures the thermal conductivity of the analyte in carrier gas, compared to the thermal conductivity of pure carrier gas. In a conventional detector at least two cell cavities are required, although a cell with four cavities is more common. The cavities are drilled into a metal block (usually stainless steel) and each contains a resistance wire or filament (so-called hot wires). They are made of tungsten or a tungsten-rhenium alloy (so-called WX filaments) of high resistance.
The filaments are incorporated into a Wheatstone Bridge circuit, the classic method for measuring resistance. A DC current is passed through the filaments to heat them above the temperature of the cell block, creating a temperature differential. With pure carrier gas passing over all four elements, the bridge circuit is balanced with a “zero” control. When an analyte elutes, the thermal conductivity of the gas mixture in the two sample cavities decreases, their filament temperatures increase, causing their resistance to

increase greatly, and the bridge becomes unbalanced – that is a voltage develops across opposite corners of the bridge. That voltage is dropped across a voltage divider (the so-called attenuator) and then all or part of it is fed to a data system. After the analyte is fully eluted, the thermal conductivity in the sample cavities returns to its former value and the bridge returns to balance, zero signal.

The larger the heating current applied to the filaments, the greater the temperature differential and the greater the sensitivity. However, high filament temperatures also result in shorter filament life because small impurities of oxygen readily oxidize the tungsten wires, ultimately causing them to burn out. For this reason, the GC system must be free from leaks and operated with oxygen-free carrier gas.

A small cell volume is desirable for faithful reproduction of peak shapes and greater sensitivity. Typically, TCD cells have volumes around 140 μL which are good for packed columns or wide bore capillaries. Make-up gas is usually required when wide bore columns are used with TCDs. The use of TCD with narrow capillaries has now become routine since cells with small volumes (e.g., 15- 20 μL) have been fabricated and several studies have shown that good capillary chromatograms can be obtained.

The carrier gas used with the TCD must have a thermal conductivity (TC) that is very different from the samples to be analyzed, so the most commonly used gases are helium and hydrogen which have the highest TC values. If nitrogen is used as a carrier gas, one can expect to get unusual peak shapes, often in the shape of a W due to partial peak inversion. The same effect occurs if one attempts to analyze hydrogen using helium as the carrier gas.