Gas Chromatography Problem Solving and Troubleshooting

Question:

My GC often requires four or more hours to stabilize after the installation and conditioning of a capillary column. This occurs for a new or previously conditioned column. I thought capillary columns required only 1–2 hours for conditioning. Is there something wrong with my GC system or column installation technique?

Answer:

Whenever a column is installed, conditioning is required to obtain a stable baseline or background signal. A column is conditioned by maintaining it at a high temperature for several hours. Usually the isothermal upper temperature limit of the column is used as the conditioning temperature. The column is fully conditioned when a stable (i.e., flat) baseline is obtained. Before a stable baseline is obtained, a sharply rising baseline followed by a slowly declining baseline is observed. Depending on the type of column, 1–4 hours may elapse before a stable baseline is obtained. It is often assumed that contaminants eluting from the column during column conditioning are from the column's stationary phase. In addition to the stationary phase, there are other possible sources of the materials eluting from the column during conditioning. Any area that is exposed to the carrier gas flow path is a possible source of these contaminants. This includes not only the column's stationary phase but also any new items or parts such as gas impurity traps, injector septa, ferrules, liners, and any part of the column residing inside the injector or detector.

It is common practice to replace septa and ferrules whenever installing a column. Upon being exposed to the high temperature of an injector or detector, contaminants are often released by new ferrules and septa. These volatile contaminants contribute to the high background observed during the initial stages of column conditioning. Sometimes conditioning the ferrules or septa requires more time than conditioning the column. Make sure your hands are clean when installing ferrules or septa. Contaminants such as lotions, soaps, or oils may be transferred to the ferrules or septa, resulting in prolonged conditioning times. Septa and ferrules can be preconditioned by baking them at an elevated temperature prior to use. Using preconditioned ferrules and septa usually results in shorter overall conditioning times and a cleaner GC system. The easiest preconditioning method is to place the septa or ferrules in a clean beaker and heat them in an oven for at least 3–4 hours. Set the oven at the same temperature as the injector or detector. Ferrules can be indefinitely maintained at the higher temperatures; septa should be stored at the higher temperatures for no more than 3–4 days. Prolonged exposure to the high temperature may cause some septa to become less resilient and increase the frequency of required replacement.

The short length of column that resides in the injector or detector (i.e., the portion above the column ferrule) can be a source of conditioning contaminants. The polyimide coating on the exterior of fused-silica columns can release a substantial amount of volatile materials for an extended time when constantly exposed to high

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temperatures. Whenever a fused-silica capillary column is installed, a short length of column is cut from the ends. Upon inserting the column into the injector or detector, a section of column that has never been in the injector or detector is now exposed to the constant high temperature of these regions. The contaminants from the new polyimide coating contribute to the conditioning background until they are fully volatilized and exit the detector. Four or more hours may be necessary before a stable baseline is obtained. If the column is removed from the injector or detector without any cutting of the column and immediately reinstalled, this problem is rarely encountered. A contribution from new polyimide only occurs when the column has been exposed to ambient air for a prolonged period. Even if the column has been held for a long time at a high GC oven temperature, any new tubing inserted into the injector or detector requires at least several hours to become free of contaminants.

Some detectors require several hours to stabilize after column installation. A longer stabilization time is required for detectors that are especially responsive to oxygen, nitrogen, or water (e.g., ECD and MS). If a column is not going to be installed within 5 min, cap off the entrance to the detector to minimize its exposure to ambient air.

If anything else was changed along with the column, additional time may be required to allow these items to equilibrate or stabilize. Injector liners and gas impurity traps are some of the more common replacable items that can contribute to the conditioning contaminants. Although the column stationary phase is a contributor to the temporary high background observed during conditioning, there are a number of other possible sources that account for a significant portion of this background. Longer conditioning times are usually required if any new parts or items are installed along with the column.