

Gas Chromatography Problem Solving and Troubleshooting

Question:

I believe the noise in my baseline is excessive. How can I determine the normal or expected amount of system noise?

Answer:

All GC systems have some noise, and whether the amount of noise is excessive or normal depends on a number of variables. Unfortunately, there is not a universal noise level that is considered to be too high for every GC system, analysis, and operating condition. Noise specifications and test conditions sometimes can be found in the GC manual. These specifications are often useful when measuring or setting acceptable noise levels.

The noise in a GC system is from several different sources. One source of noise is the contribution from the mechanical and electrical components of the detector and data system. These include such items as electrometers and A/D converters. The functional detector also contributes to it. This is the noise generated when the detector is operational (i.e., heating of the detector, proper gas flows, flame lit, current applied). Another possible contribution is from the carrier gas. The final source is the column. There are a series of steps that can be used to measure the amount of noise contributed by each area.

To determine the amount of mechanical and electrical noise, seal the detector entrance with a leak-free cap or plug. Make sure that the detector is turned off (i.e., without any heating, gas flow, current, lamp). The data system or integrator should be set to the values used in a typical analysis. Plot the signal for about 10 min. The baseline should be flat with an extremely low amount of noise. Very sensitive settings may be necessary to see the baseline noise. This is the noise contributed by the electrical and mechanical components of the detector and data system. Baseline drift and excessive or higher than normal noise in this area is an indicator of an electronic problem such as a faulty electrometer, loose or incorrect connections, or a bad cable.

The noise due to the operational detector is determined by turning the detector on without a column installed. Seal the detector entrance, heat the detector to its typical operating temperature, supply all detector gases, including the makeup gas at the recommended flow rate, and turn on any filaments, lamps, or flames. Allow the detector to stabilize for at least 15 min. Some detectors may require a longer time for stabilization. Plot the baseline for about 10 min. The baseline should be flat, with a noise level higher than the mechanical and electrical noise level. The additional noise is contributed by the detection process. Excessive or higher than normal noise in this area is an indicator of a dirty detector or gases, an incorrect detector gas flow rate, a leak in the detector or a detector gas line, faulty or expired filaments, beads, lamps, etc., or a loose or corroded connection between the detector and the electrometer.

Determination of the contribution of the carrier gas to the noise level requires several meters of deactivated fused-silica tubing (tubing without any stationary phase) for a capillary column system or a clean, empty packed column for a packed-column system. Install the fused-silica tubing or empty column in the injector and detector in the same manner as the regular column. Supply carrier gas at the same flow rate as used for a typical analysis. Sometimes the flow rate is too high, even at the lowest head pressure settings, when an empty packed column or short lengths of fused-silica tubing is used. Use long and narrow tubing or empty columns to obtain a higher head pressure so that the proper flow rates can be set. In any case, set the carrier gas flow rate as close to the typical flow rates as possible. Condition the tubing or empty column for 15–30 min at the usual conditioning temperature. Plot the baseline for about 10 min with the oven set at 40–50°C. Any additional noise above that measured for the operational detector is the contribution of the carrier gas. Impure carrier gas, impurities picked up by the carrier gas (e.g., from gas lines, traps, regulators), or a leak in the carrier gas line are the usual causes of excessive or higher than normal noise in this case.

The column contribution to the noise is determined by installing and conditioning the column in the usual manner. All of the GC conditions should be set as for a typical analysis. Plot the baseline for about 10 min with the oven set at 40–50°C. A noise greater than that measured in the carrier gas step is the noise contribution from the column. Most of this noise is from column bleed (i.e., normal stationary phase degradation). Excessive or higher than normal noise is an indicator of a contaminated or severely damaged column.

If an empty packed column or deactivated fused-silica tubing is not available, the noise contribution from the carrier gas cannot be determined since that step has to be skipped. If the carrier gas step is omitted, the additional noise in the system after column installation is the sum of the carrier gas and column contribution. It is not possible to determine the individual contribution from the column or carrier gas. An advantage of measuring the carrier gas contribution to the noise level is that additional information can be obtained when the empty column or tubing is installed. Some detectors are sensitive to changes in the carrier gas flow rate. This may appear as baseline drift that corresponds with the temperature program. By running a blank (i.e., analysis without an injection) with the empty column or tubing installed, any baseline drift caused by the normal change in the carrier gas flow can be measured. If a baseline drift problem occurs, the amount of drift due to the carrier gas flow rate change is known.

The purpose of *Chromatography Problem Solving and Troubleshooting* is to have selected experts answer chromatographic questions in any of the various separation fields (GC, GC-MS, HPLC, TLC, SFC, HPTLC, open column, etc.). If you have questions or problems that you would like answered, please forward these to the *Journal* editorial office with all pertinent details: instrument operating conditions, temperatures, pressures, columns, support materials, liquid phases, carrier gas, mobile phases, detectors, example chromatograms, etc. In addition, if you would like to share your expertise or experience in the form of a particular question accompanied by the answer, please forward to JCS Associate Editor, *Chromatography Problem Solving and Troubleshooting*, P.O. Box 48312, Niles, IL 60714. All questions/answers are reviewed to ensure completeness. The *Journal* reserves the right not to publish submitted questions/answers.

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Associate Editor