

## Liquid Chromatography Problem Solving and Troubleshooting

### Question:

I have enclosed a chromatogram (Figure 1) that illustrates what I recently have been observing in a random fashion over the last several weeks. Luckily, the problem baseline has not interfered with my analyses yet. A colleague has suggested that my column is deteriorating, but before I purchase a new column, I would like you to comment.

### Answer:

The situation that you describe may not be a column problem. It may indeed be a problem with the detector lamp, but it is important to isolate the most probable cause of the erratic baseline before you start replacing items. Because you mentioned that the observation was recently noticed, it is important to determine if the onset of the problem was coincident with any change in mobile phase, solvent (or lot) use, plumbing activity, repairs, etc. When the baseline drops as it does in the chromatogram (see region by arrow on right side), it is often but not always caused by a new mobile phase that, having equilibrated with the column, elutes from the column. If the answer is that nothing changed, I would suggest considering all of the features of the chromatogram shown in Figure 1.

The chromatogram has several characteristics that should be discussed. In addition to an apparent jump in the baseline, there is, as you mentioned, occasional random noise appearing during the run (see arrow pointing to region in the lower box). Do some quick tests to eliminate the possibility that changes in the flow rate are the cause. Examine the pressure trace to determine whether a variation in pressure is associated with the noise and/or the drop in baseline. If there is a relationship, the pump or injector must be repaired or cleaned to establish a constant flow.

If the flow is constant, this brings us back to my original suggestion: the detector lamp. When both types of baseline upsets occur together in the same chromatogram, the cause is often a lamp that has served its useful lifetime. The lamp is weak and/or is dying. With a weak lamp as a hypothesis, check the lamp voltage and look up when the lamp was installed. Many lamps have a lifetime of one year. Other observations that support this hypothesis are (a) the quick onset of periodic baseline noise spike as well as the sudden subsiding of these ticks and (b) the very erratic noise between the spikes (shown in the top box).

Although it is tempting to quickly conclude that the lamp is the cause, it is important to do some diagnostic tests and investigations before the final cause is identified and corrected.

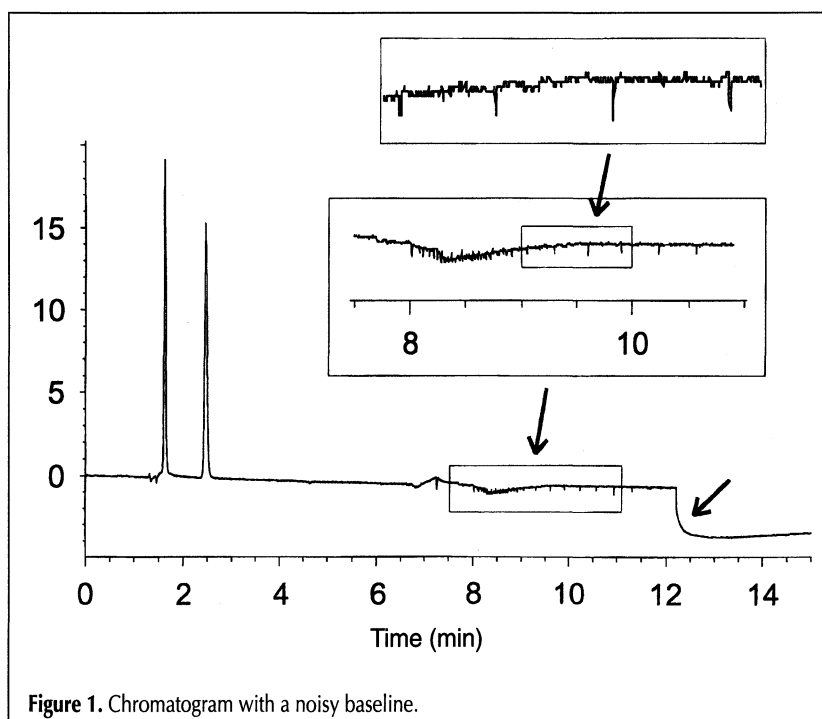


Figure 1. Chromatogram with a noisy baseline.

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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