

## ***Gas Chromatography Problem Solving and Troubleshooting***

### **Question:**

I get a slightly different value every time I measure the carrier gas flow rate for my capillary column. I am measuring the flow rate out of the detector using a bubble flow meter and helium as the carrier gas. Is it normal for the flow rate to change, and how do I determine which flow rate is correct?

### **Answer:**

The average linear velocity is a better measure of the impact of the carrier gas on the chromatographic results. However, there are situations in which the carrier gas flow rate is a necessary measurement. Unfortunately, it is difficult to obtain accurate and precise carrier gas flow rate measurements for smaller diameter capillary columns using a flow meter. Helium carrier gas flow rates for 0.20–0.32-mm-i.d. columns are typically 0.8–2 mL/min. Higher carrier gas flow rates such as those used with 0.53-mm-i.d. columns can be accurately measured using a flow meter. The types of flow meters used with GC systems are not very accurate at such low flow rates. Bubble flow meters using a soap solution are inaccurate due to the variable water content in the measured gas. Even the newer digital flow meters are often inaccurate at very low flow rates. Small movement of the tubing leading into the flow meter can result in a positive flow reading, even when there is no gas flow. The typical 0.1–0.3 mL/min of artificial flow is a significant portion of the total flow rate for a capillary column. Fluctuating or incorrect flow rates are obtained unless the tubing is held completely motionless.

To measure the carrier gas flow rate, the flow meter is connected to the exit of the detector. This is a potential source of several errors or problems. The connection between the detector and the flow meter tubing must be free of leaks. A leak in this area leads to a measured flow rate that is lower than the actual flow rate. Each time the flow meter is attached, there is potential for a different size leak to occur. Obviously, this leads to measurement differences and errors. Due to the design of some detectors, it is difficult to obtain a leak-free seal without a special adapter, usually supplied by the GC manufacturer. Sometimes an adapter has to be made in the laboratory. Make sure to use materials that are stable at temperatures of 300–400°C. Due to their design, some detectors have multiple places through which small amounts of gas can escape. Close inspection of the detector is required to determine if there are other possible flow paths other than the detector exit where the flow meter is connected.

Turning off all the other detector gases when measuring the carrier gas flow rate with a flow meter is recommended. Measuring the carrier gas flow rate by difference ( $[\text{detector gas flow rate} + \text{column carrier gas flow rate}] - \text{detector gas flow rate}$ ) is not recommended. The total gas flow rate for some detectors is 300–500 mL/min. Trying to measure a difference of 1–2 mL/min at these high flow rates is inaccurate. With older or abused instruments, one or more of the detector gas flow controllers or pressure regulators may no longer shut off completely. There is still some flow of gas through the controller or regulator when it is in the off position. Measurement errors will occur if adjustments for this spurious gas flow are not made.

Other difficulties are sometimes encountered when using a flow meter for measuring carrier gas flow rates. Carrier gas flow rate is dependent on column temperature. If the same flow rate is set at a different column temperature, a different flow rate is obtained when the column is returned to the original temperature. Differences in retention times and peak resolution are obtained. Turning off all the detector gases for some detectors is not recommended. Sometimes minutes or even hours are needed for the detector to stabilize after a large change in the detector gas flow. NPDs, TCDs, and especially ECDs are known to exhibit this type of behavior. The process of properly measuring the carrier gas flow rate may render the GC unusable for up to several hours. Some detectors, most notably the MS, do not have a readily accessible exit port where the column flow rate can be measured. Using a flow meter is not possible with these types of detectors. Whenever a flow meter is used, the carrier gas flow rate at the exit end of the column is obtained. The carrier gas flow rate is not the same throughout the length of the column. It is faster at the rear of the column than at the front. The flow rate measured with a flow meter is not the carrier gas flow rate inside or at the front of the column, but the carrier gas flow rate at the exit end of the column. The flow rate at the front or inside of the column has the largest influence on the chromatography (e.g., retention time, resolution, peak shape). While good and reasonably repeatable chromatographic results can be obtained when setting carrier gas flow rates using a flow meter, there is a better and more reliable method.

**Table I. Recommended Nonretained Compounds**

Detector	Compounds
FID	methane, butane*
MS, TCD	methane, butane*, air
ECD	SF <sub>6</sub> , methylene chloride†
NPD	acetonitrile‡

\* Easily obtained from a disposable lighter.  
† Do not inject liquid solvent. Inject 1–2 μL of the headspace from a sealed vial containing 5–10 drops of methylene chloride. Use a column temperature of 40°C or higher.  
‡ Do not inject liquid solvent. Inject 1–2 μL of the headspace from a sealed vial containing 5–10 drops of acetonitrile. Use a column temperature of 90°C or higher.

An average flow rate can be determined without the use of a flow meter. All that is needed is a nonretained compound for the column being used. A list of nonretained compounds can be found in Table I. An injection of 1–2 μL of the appropriate compound using a split injector is recommended. After the retention time for the injected compound is obtained, the average flow rate can be calculated using Equation 1 ( $r$  is the column radius in centimeters,  $L$  is the column length in centimeters, and  $t_m$  is the retention time of the nonretained compound in minutes).

$$\bar{F} = \frac{\pi r^2 L}{t_m} \quad \text{Eq 1}$$

If the flow rate is too high, the head pressure needs to be decreased; increase the head pressure if the flow rate is too low. Repeated injections and adjustments are necessary until the desired flow rate is obtained. A slightly different flow rate is obtained than that measured with a flow meter because the average flow rate is obtained instead of the flow rate at the end of the column. Calculating the average flow rate provides a better and more reproducible measure of the carrier gas flow rate.

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.

Dean Rood  
Associate Editor