Gas Chromatography Problem Solving and Troubleshooting

Question

Using a nonretained compound and the right formula, I obtain a different carrier gas average linear velocity than the one reported by the GC's electronic pressure control system. Also, the carrier gas flow rate is different than the one I measure with a flow meter. Why are the values different, and which ones are correct?

Answer

Electronic pressure control systems use a series of equations to calculate and adjust the column head pressure to obtain the desired carrier gas velocity or flow rate. They do not directly measure the carrier gas velocities or flow rates, but set a column head pressure based on several GC and column parameters. The type of carrier gas, column length and diameter, and oven temperature are the important parameters. The type of carrier and column dimensions are provided by the user. The column temperature is obtained internally by the system. Discrepancies in any of these values will lead to inaccurate average linear velocity or flow rate values being set and reported by the electronic pressure control system. Flowmeters may also be inaccurate if used improperly. The combination of flowmeter and electronic pressure control system inaccuracies is probably responsible for the different flow and velocity values.

Electronic pressure control systems require periodic calibration. If the system is out of calibration, inaccurate velocity and flow rates are obtained. User-performed calibrations are usually possible, but a qualified service person may be required for some GCs. Because oven temperature is also an important variable, oven accuracy impacts the carrier gas flow and velocity values. Ovens may also require periodic calibrations. Column length and diameter is input into the electronic pressure control system. Usually, the nominal values are used instead of the actual length and diameter values. If the actual length or diameter is different from the nominal values, flow or velocity errors may occur. For example, an actual column length of 29 m input as 30 m would result in an error of approximately 3% in the average linear velocity. Actual column diameters may vary by ± 0.01–0.03 mm (for 0.25–0.53-mm i.d. columns) from the nominal values. Diameter discrepancies can lead to velocity errors of 8% or more. The combination of column length and diameter discrepancies may result in velocity and flow differences of 12% or more between the actual and set values. Shorter column lengths and larger diameters are the most susceptible to discrepancies between the actual and set values. Out-of-specification oven temperatures or pressure control systems also add to potential inaccuracies.

Manually measuring and setting the average linear velocity requires injecting a nonretained compound and determining its retention time. The length of the column (in centimeters) is divided by this retention time (in seconds). This provides the average linear velocity in the desired units of centimeters per second (cm/s). As mentioned before, if the length of the column used in the calculation is incorrect, an inaccurate velocity is obtained. Inaccuracies in the column diameter are inconsequential, because diameter is not used in the average linear velocity calculation. Column temperature affects average linear velocity; thus, the same oven temperature needs to be used when comparing average linear velocities or flow rates.

Obtaining accurate flow rates for capillary columns using a flowmeter can be difficult. Typically, capillary column flow rates are 1–4 mL/min, whereas packed column flow rates are 10–40 mL/min. Most flowmeters can accurately measure the higher flow rates of packed columns but have accuracy difficulties with the lower flow rates of capillary columns. Even if the detector gases are left on and the carrier gas flow rate is measured by difference (i.e., total gas flow from the detector with and without the carrier gas present), inaccuracies occur. Most flowmeters measure volumetric flow rates, which are dependent on gas pressure and temperature. Electronic pressure control systems often report mass flow rates compensated for pressure and temperature. Mass and volumetric flow rates are not equal except at one specific set of conditions (STP). Bubble flowmeters are inaccurate due to the amount of water vapor in the measured gas (from the soap solution used to create the bubbles). Digital and bubble flowmeters often give slightly different flow

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values. Finally, it is sometimes difficult to get a good seal between the flowmeter tubing and the detector housing, and some detectors have many sites for exiting gases to escape.

The carrier gas average linear velocity and flow rates set and reported by electronic pressure control systems are based on calculations and not direct measurements, and they are dependent on the accuracy of the various parameters. Average linear velocities obtained by injecting a nonretained compound are primarily dependent on the correct column length and less affected by the other variables. Measuring flow rates using flowmeters are subject to many potential pitfalls and sources of inaccuracy. There are a number of areas where errors can be introduced into the average linear velocity or flow rate values. Careful equipment calibration and measurement of column dimensions is required to obtain accurate and comparable values. Fortunately, precise carrier gas measurements are more critical than accurate ones. While accuracy is still important and desired, more problems probably arise from erratic carrier gas flows and velocities than slightly inaccurate ones.