Gas Chromatography of Hydrocarbons & Chromatography Basics

OVERVIEW

Prior to the invention of gas chromatography in the 1940s and 50s, the separation of small amounts of close-boiling volatile liquids was very difficult. Gas chromatography is a simple, versatile method for the separation and analysis of volatile mixtures. The method involves injection of a small amount of sample into a moving stream of gas (mobile phase or carrier gas) that is passed through a column (stationary phase). Separation of a mixture into its individual components is achieved if the components are retained in the column to varying extents. The time it takes for a component to exit the column, the retention time (t_R), may be used for qualitative analysis.

In this experiment hydrocarbons will be studied with respect to retention time. From this data basic chromatographic figures of merit such as retention factor, selectivity factor, number of theoretical plates and plate height will be determined. These parameters are discussed in Chapter 26 of the text. In addition an unknown mixture of hydrocarbons will be qualitatively identified.

MATERIALS/METHODS

1% stock solutions of n-pentane, n-octane, n-nonane, n-decane, and n-dodecane dissolved in $CHCl_3$ are provided, as well as unknown mixtures containing 2 or more of these hydrocarbons. A mixture of all hydrocarbons in $CHCl_3$ is also available for a temperature programmed run.

A 10 µl syringe for sample injection should be used. Be sure to rinse the syringe well with $CHCl_3$ between injections, as the GC is very sensitive. Inject approximately 0.5 µl of sample. First pull the plunger out part way to fill the syringe with air, then take about 0.5 µl of sample, and finally pull in more air. You should have a sample of liquid "sandwiched" between air. When injecting the sample, rapidly (but gently so as not to bend the fragile tip) inject the sample immediately after piercing the septum of the inlet in the GC.

Although the GC should be started up for you, make sure the data acquisition parameters are set up correctly. See my web site for instructions on how to use the GC, there are usually printed instructions near the GC as well. Some instructions specific to these separations are as follows:

All isothermal runs with an oven temperature = 60°C
Temperature programmed run: 4 min @ 60°C, ramp rate of 20°C/min to 160°C, hold for 1 minute.

You can stop a run when you believe all of your components have exited the GC, you do not have to wait until the program stops collecting data on its own. However be sure all components have exited the GC, it is very confusing if components from a previous run exit the GC during a subsequent separation.

PROCEDURE

Here is a list of things to accomplish in approximate recommended order.

1. Make multiple injections of $CHCl_3$ until you are confident the chromatogram can be repeated and you know its retention time.
2. Inject the individual hydrocarbons in $CHCl_3$ to find their retention times. Use the retention time for pentane as the dead time, t_M.
3. Obtain a chromatogram of your unknown.
4. Obtain a chromatogram of the mixture of hydrocarbons.
5. Obtain a 2^{nd} chromatogram of the hydrocarbon mixture using the temperature program detailed in the previous section.

Save all of your chromatograms. This data can be worked up either on the computer as is, or exported as a text file for use off-line in a spreadsheet. The latter is recommended since you may want access to this data as you are writing your report.

DATA TO INCLUDE FOR YOUR REPORT

1. Retention times for each hydrocarbon
2. The retention factor for each hydrocarbon
3. The resolution between nonane and decane
4. The number of theoretical plates for nonane & decane
5. The plate height for nonane & decane
6. Any chromatograms/illustrations, etc. to support the data above, and the discussion.

WHAT TO ADDRESS IN YOUR REPORT

- How did you determine the identity of your unknown?
- What is the effect of temperature programming on the two chromatograms obtained – and why?
- Explain the trends of retention time and retention factor for the hydrocarbons
- How is the resolution between these hydrocarbons – good, bad, indifferent?
- Discuss the H and N for nonane and decane. Are these values about what you would expect given your reading in the text? Are they the same for each hydrocarbon? Why or why not?