Chemistry 365
Gas Chromatography

Gas chromatography (G.C.) is a powerful method for both qualitative and quantitative analysis of volatile compounds. Numerous applications of chemical analysis by G.C. have been published. For example: analysis of hydrocarbons in fuels, determination of gas-phase organic pollutants, determination of VOCs in water, measurement of pesticides in soils, fragrance analysis, and flavorant analysis. To date, nearly 40% of all separations are carried out by G.C. The separation process, carried out in the gas phase inside a column, is primarily based on differences in molecular weight and boiling point. Quantitative analysis can be accomplished through the use of a variety of detectors including thermal conductivity, flame ionization and mass spectrometry. Identification of the components in a mixture is carried out by comparing the retention times of the components in the mixture with the retention times of pure, known samples (standards). Additional qualitative information may be obtained using a mass spectrometer. Under constant column temperature, injection temperature, and carrier gas flow rate, a particular compound will exhibit exactly the same retention time each time it is injected, regardless of whether it is in a pure form or in a mixture (unless somebody changes the column!). Relative amounts of each component in a mixture can be obtained by measuring the peak area for each component in a mixture of known composition and comparing these peak areas to the peak areas of an unknown mixture. In other words, peak area is proportional to concentration.

Your mission for this lab is to separate a mixture of hydrocarbons using a gas chromatograph equipped with a capillary column and a flame ionization detector or a mass spectrometer. Most gas chromatographs are capable of regulating the temperature of the column and the injector temperature and give the operator infinite control over the flow rate of helium or nitrogen - the carrier gas - through the column (typical flow rates range from about 2 mL/min. to 80 mL/min.). You should optimize the separation of a mixture of hydrocarbons using a standard solution containing a known weight percent of at least three compounds (see the procedure below for making this standard). Consult with your instructor prior to making up your standard. When preparing your standard solution, try to have roughly equal amounts of each of the components. This makes the optimization of the separation conditions a heck of a lot easier!

Following optimization of the separation parameters, you will be given an unknown. Your mission is to determine which compound(s) is/are present (e.g. qualitative analysis rather than

quantitative analysis). If you wish to receive extra credit, you may bring a gasoline sample in for analysis. The gasoline sample (1-50 mL) should be collected in a glass vial.

So, to get started, do the following

1. Form a research group
2. Design the most efficient and information-rich strategy you can to investigate the effects of changing the analytical variables (described above) to achieve the best separation (or resolution; a definition of this term is given on page 511 of your textbook) of your compounds.

PROCEDURE

1. **Sample preparation**: Prepare a stock standard of n-alkanes (1 mg/mL) including n-octane, n-decane and n-dodecane in pentane. Dilute the stock solution in pentane to a concentration of 100 μg/mL of each compound. If you wish to analyze a gasoline sample, dilute it 1:10 in n-pentane.

2. **Instrument settings:** The temperature of the column oven should be set to about 50˚C (depending on what compounds you are injecting!). *If you know the boiling point of your analytes*, you can use this information to set up program which varies the temperature of the column oven during the time-course of the separation. This is known as temperature programming and, when done properly, will dramatically reduce the analysis time. The temperature of the injector port and the detector should be set to about 250˚C. Be careful to record all the operating conditions in your lab notebook.

3 **Running samples**: Using the auto-sampler, inject a 1 μL sample of your standard into the column. The first peak to elute from the column is the compound with the lowest boiling point/molecular weight. In what order do the remaining components in your mixture elute? Why? If you have any doubts about the order of elution, inject pure samples of any the components and compare the retention times of the pure samples with those of the standard mixture.

4. **Data analysis:** Accurately determine the retention times of each component. Determine peak areas. What does this tell you?

5. **Unknown analysis:** Obtain a chromatogram of an unknown sample. Determine the retention times of the peaks in your unknown. Identify the component(s) present in your sample. Integrate the peak areas. Compare the peak area(s) from the unknown to your standard and estimate the concentration of each component in your unknown.