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 retention times of the components in the mixture with 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 identify the hydrocarbon(s) present in an unknown using a gas chromatograph equipped with a capillary column and a flame ionization detector or a mass spectrometer. Identification of the hydrocarbon(s) can be accomplished by comparing the chromatogram obtained from a standard mixture to the chromatogram of your unknown. 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.

PROCEDURE

1. **Sample preparation**: Prepare a standard of n-alkanes including n-octane, n-decane and n-dodecane in pentane by placing 1-2 drops (from a disposable pipet) of each n-alkane into a GC autosampler vial. Fill the vial with n-pentane until the vial is ¾ full. If you wish to analyze a gasoline sample, dilute 1 drop of gasoline with n-pentane in an autosampler vial (again, fill the vial ¾).

2 **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.

3. **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.