Gas Chromatography

I. Introduction

Gas chromatography is a powerful analytical technique for the separation and identification of complex organic mixtures. A mixture is injected into the gas stream of the GC and the components separated by virtue of their different affinities for the column packing. A wide variety of components can be separated, even those possessing very subtle differences, such as geometrical and optical isomers of the same substance. In this experiment, some of the separation parameters available in GC analysis will be evaluated and an unknown mixture of straight chain hydrocarbons will be analyzed. Since it is very difficult to reproduce the amount of sample injected into the gas chromatograph (a sample loop like in the HPLC experiment is not used), an internal standard will be used to allow quantitative determination of the components in your unknown. Temperature programming will be used to provide an efficient separation of all components in the injected sample.

II. Equipment and Materials

A. Equipment

1. Shimadzu Model 17A gas chromatograph with flame ionization detector.
2. J&W DB-5 capillary column (15 m x 0.25 mm x 0.25 μm).
3. 5-μL hypodermic syringe with pointed needle.
4. 1.0-mL and 0.5-mL class A glass transfer pipets.
5. Two 2-mL glass vials plus caps.

B. Solutions (obtain unknown from instructor)

1. Absolute ethanol.
2. GC standard mixture (1.00% each of octane and nonane in ethanol).
3. Unknown mixture containing octane and nonane in ethanol.
4. Internal standard solution (2.00% decane in ethanol).

III. Procedure

A. Sample Preparation

1. Clean two 2-mL vials with ethanol and allow to dry.

2. Pipet exactly 1.0 mL of the alkane standard solution into a clean vial, add exactly 0.5 mL of the decane internal standard solution, cap and mix well.

3. Pipet exactly 1.0 mL of the your unknown solution into a clean vial, add exactly 0.5 mL of the decane internal standard solution, cap and mix well.

B. Quantitative Analysis of an Unknown Mixture

1. Your instructor will demonstrate how to inject samples into the gas chromatograph and how to collect and print data using the computer interface.

2. Make sure that the method **321is2** is loaded. This method will initially operate the column at a temperature of 70°C for 1.5 minutes, then ramp the temperature at a rate of 30°C/min until it reaches 100°C and hold it there for 0.5 minute. The total run time is 3.0 minutes, and the carrier gas flow is 1.0 mL/min. Prepare the GC for injection and inject approximately 1-µL of the spiked standard solution. Record the name of the file where the data are saved. Print out a copy of the chromatogram and a report listing retention times and peak areas for all of the components. Identify each peak in your chromatogram.

3. Make at least two additional injections of the spiked standard solution and print out the chromatogram and report for each run. Assess the reproducibility of your measurements by calculating the ratio of the octane peak area to the decane peak area for each injection. This ratio should vary by only 2-3%.

4. Make several injections of your spiked unknown solution using the same approach used for the standard in B.2 above. Obtain printouts of the chromatogram and report for each run.

IV Calculations

A. Column Efficiency (N)

Using data from the standards run in III. B, calculate the number of theoretical plates (N) for octane using:

$$N = 16\left(\frac{t_R}{W}\right)^2$$

where W is the magnitude of the base of the triangle defining the octane peak. Make sure that the units of t_R and W are the same.

B. Unknown Mixture

Multiple injections of a sample will usually result in different peak areas for a given component because it is difficult to reproduce the injection volume. These variations are compensated for by adjusting (normalizing) the component peak area by dividing it by the peak area of the internal standard (decane). In this way the concentration of each component in your unknown can be determined by a simple comparison of normalized peak areas:

$$\frac{\left(\frac{\textit{Component AREA}}{\textit{Decane AREA}}\right)_{unk}}{\left(\frac{\textit{Component AREA}}{\textit{Decane AREA}}\right)_{std}} = \frac{C_{unk}}{C_{std}}$$

where (Component AREA/Decane AREA)$_{unk}$ is the normalized peak area for a particular component in the unknown solution, (Component AREA/Decane AREA)$_{std}$ is the <u>average</u> normalized peak area for a particular component in the standard solution, and C_{unk} and C_{std} are the unknown and standard concentrations of that component, respectively. Report the mean concentration and its RMD for each component of the unknown. The precision of your results should be 2-3%.