Gas Chromatography
Detector Response to Hydrocarbons
and Analysis of Gasoline

The flame ionization detector (FID) used in gas chromatography (GC) is quite sensitive to carbon compounds which do not contain electronegative species. Simple hydrocarbons fulfill this condition. The purpose of this experiment is to optimize the Agilent Technologies 6890 GC for the separation of a hydrocarbon mixture, investigate the purported sensitivity of the FID in some detail, and identify some hydrocarbons in a sample of gasoline.

1. **Optimization of Column temperature and split ratio**.

a. <u>Sample preparation</u>:

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 concentrations of 500, 250, 125, 100 and 50 μg/mL of each compound.

b. <u>Setting initial instrument parameters</u>

Set the injector temperature to 250°C and the FID detector to 250°C using the appropriate buttons in the ChemStation software. These conditions will remain constant throughout your analyses. Oven temperature(s), carrier gas flows, and the split ratio may also be adjusted using the ChemStation software.

Initial conditions of column temperature should be around 70°C. You should look up physical properties like boiling points and molecular weights of each component in your mixture in the CRC Handbook as a guide to initial operating conditions. It is probably wise to run your first round of optimization runs at a constant column temperature rather than trying to use temperature programming.

You will need to determine the optimum split ratio too. When the column oven has reached its initial temperature, inject a 0.5 μl sample of your hydrocarbon mixture. If the peak(s) are off scale or not Gaussian, increase the split ratio. If your peaks are too small (less than 10% of full scale), increase the size of your sample or decrease the split ratio.

Your mission in this part of the experiment is to get all of your components resolved in the shortest period of time without sacrificing peak shape. The HP-5 column that is installed in the chromatograph separates materials on the basis of their molecular weights. Selection of three or four components with significantly different molecular weights will make this optimization easier. You should also identify which peak is due to which component. If you have any doubts, run a pure sample of one or more of the components after you have optimized the separation. Since the retention time of any compound is constant at constant conditions whether it be a pure sample of the compound or present in a mixture with other materials, you should be able to unequivocally identify each of your components in the chromatogram. ***Report retention times of all components, as well as the precise conditions required to obtain your optimized chromatogram***.

2. **Investigation into the sensitivity of the FID**

Obtain good (pretty) chromatograms of each of your standard solutions. Obtain peak areas for each of the five components. The ChemStation software has an integration function which will provide you with a number proportional to the peak area of each peak for each sample you run.

Prepare a table that gives both the weight and the number of moles of each of your four components in each sample.

For each hydrocarbon, plot a calibration curve with peak area on the y-axis and amount of analyte in moles on the x-axis. Also, for each hydrocarbon, plot a calibration curve with peak area on the y-axis and amount of analyte in grams on the x-axis. Estimate sensitivity for each of the four hydrocarbons. Is the peak area correlated with the number of grams of analyte or with the number of moles of analyte? Discuss.

Next, for the first solution:
1) construct a plot of peak area vs. #carbon atoms
2) construct a plot of log #carbon atoms vs. peak area
3) construct a plot of retention time vs. #carbon atoms
4) construct a plot of log retention time vs. #carbon atoms.

Finally, construct a table that lists, for each analyte, T_r, W_b, N and H. Be sure you are consistent with units for T_r and W_b.

3. **Gasoline Sample**

Run a sample of gasoline under the conditions you used for optimum resolution. Identify as many peaks as you can by using the retention time plots obtained above.