EXPERIMENT 9
GAS CHROMATOGRAPHY QUALITATIVE ANALYSIS USING PACKED COLUMN

The purpose of this experiment is to identify the normal alcohols which are present in an unknown mixture. The student will also become familiar with the use of a temperature programmable gas chromatograph.

See also the gas chromatograph and integrator instruction sheets.

Procedure

A mixture of known alcohols will be provided to run on the gas chromatograph so that the \( t_r \) values may be obtained.

Instrument Start Up.

Turn on the gas chromatograph following the directions which pertain to the type of instrument you are using. There are slightly different directions for each manufacturer's gas chromatograph. We will be using a HP 5890 gas chromatograph.

The following are the start up instructions. Steps #1 to #6 will normally have been done for you, since, while the class is doing this experiment, the gas chromatograph is left on day and night.

1. Insert a suitable column, such as Carbowax-20M, into the chromatograph.
2. Loosen the diaphragm valve on the nitrogen cylinder.
3. Turn on the main cylinder valve.
4. Adjust the cylinder diaphragm valve to read 30-40 p.s.i.g.
5. Ignite FID.
6. See HP5890 gas chromatograph Operating Instructions' sheets.

You will need to make 2 groups of injections into the gas chromatograph.

1. Inject a 1 microL sample of a known mixture of linear alcohols (from ethanol to octanol). You should see all seven peaks of alcohols in your printout. From your printout, determine capacity factors for each of components of your mixture as well as the number of theoretical plates in your column. Optimize the program of your instrument in order to have the appropriate resolution in 10 min. (This optimization is already done for you).

**Note the following:**
1. After piercing the septum with the needle and pushing the needle quickly all the way in, depress the plunger QUICKLY to insure that the whole sample is injected at one time. As soon as the needle tip is through the septum, liquid sample boils out in the hot injection port.
2. Remove the needle with a rapid straight outward motion, taking care not to bend the needle.
3. The difference between the time of emergence of the methane peak and a compound peak is the corrected retention time of the compound.
4. Before injecting a new sample, rinse the syringe about 10 times with ethanol then 10 times with the new sample to rinse the ethanol out of the syringe.
5. Keep your unknown tightly capped at all times to prevent evaporation of your mixture.
6. This entire experiment must be run at one time. You cannot run your standards one day and unknowns another day since retention times are not exactly repeatable over a long time period.

Quantitative determination of the mixture.
Take 5 mL of your unknown sample and add 5 mL of methanol. Mix the resulting solution carefully. Inject the portion of solution at the optimal conditions.
Take another 5 mL of your unknown sample, add 0.10 mL of pentanol and 4.90 mL of methanol. Now you have the same solution as before but with a 0.100 mL spike. Inject the same amount as before at exactly same conditions. Calculate the amount of pentanol you had had in your unknown solution using the combination of internal standard and standard addition methods. Use butanol peak as your internal standard.
Do the same determination for other alcohols in a mixture; for example, for hexanol and heptanol.

Hints:

1. In order to make an internal standard correction using the butanol peak, you must divide all peak areas you received by the peak area of a butanol peak. Use this numbers ($I_x$, $I_{spike}$, etc.) for further calculations.

2. Use volume per cent (%%(v/v)) as your concentration unit.

$$v_x = v_{spike} \times \frac{I_x}{I_{spike} - I_x} = 0.10mL \times \frac{I_x}{I_{spike} - I_x}$$

$$\%\%(v/v) = \frac{v_x}{v_{total}} = \frac{v_x}{5mL} \times 100\%$$

Determine selectivity factors.