EXPERTIMENT 10
GAS CHROMATOGRAPHY QUANTITATIVE ANALYSIS USING CAPILLARY COLUMNS

The purpose of this experiment is to identify the aromatic hydrocarbons which are present in an unknown mixture and to determine the composition of the mixture. The student will also become familiar with the use of a temperature programmable gas chromatograph using capillary column and splitting injection mode.

> See also the HP 5890 gas chromatograph instruction sheets and a tutorial for ChemStation software (Computer Lab No.2).

Procedure

A mixture of hydrocarbons (xylenes) will be provided to run on the gas chromatograph so that the retention time (t_r) values may be obtained.

Prepare a solution of (given to you as an unknown) xylenes in cyclohexane by ten-fold dilution in a 10 mL volumetric flask.

Take 1.00 mL of the analyte, and add cyclohexane to the mark.

Inject a 1 μL sample of this solution using autosampler. Record the chromatogram.

Using the standard solutions of individual components, make the assignment of each peak in chromatogram.

Take 1.00 mL of o-xylene, and add cyclohexane to the mark. Inject a 1 μL sample of this solution using autosampler. Record the chromatogram. Repeat it for ethylbenzene, m-xylene and p-xylene.

Data analysis.

Determine the composition of unknown mixture using the internal normalization method in %%. Compare your results with standard addition.

Hint*: All xylenes have exactly the same brutto formula C_8H_{10}. Intensity of FID signal is practically the same for all compounds of the same formula.*

Compare your GC results with those from Raman spectroscopy experiment and IR spectra (experiments 7 and 9).

Calculate capacity factors, separation factors, and distribution coefficients for xylenes.