

Summary Report

The objective of this lab was to determine the composition of hydrocarbons in an unknown mixture prepared by the lab instructor. In order to do that, a gas chromatography was done on both known and unknown mixtures of hydrocarbons. The mixtures of hydrocarbons were separated into the column of the gas chromatography (GC) operation machine. The column was coated with high-boiling liquid, known as the stationary phase. The volatile hydrocarbons interacted with the stationary phase depending on their structure. Less interaction would lead to a faster elution through the column. The exiting gas at the end of the column was analyzed by passing through a detector. The data were recorded and plotted in Excel. The retention time (the time taken for the component to pass through the column) was calculated by referring to the plot of chromatography of each hydrocarbon. 1 microliter of each type of hydrocarbon (Heptane, Cyclohexane, Toluene and p-Xylene) was injected into the GC operation machine. Based on each chromatogram plotted in excel (**Figure 1 – 4**), the retention time was the time taken for the sample to reach the peak of the curve from the injection point. In the order of **Heptane, Cyclohexane, Toluene, and p-Xylene**, the retention time was **86, 96, 136 and 182 seconds**, as tabulated in **Table 4**.

A mixture of hydrocarbons was prepared by weighing a desired amount of each hydrocarbon, and was called as a ‘known mixture’. The mass of each hydrocarbon was recorded, as tabulated in **Table 2**. By using the ‘known mixture’, three trials of chromatography were obtained by injecting 1 microliter of sample into the GC machine per trial. The data for each sample were collected, as shown in **Table 1**, and the chromatography was plotted, as shown in **Figure 5**. The second trial of known mixture was rejected due to the fact that it did not have the same consistency in values like the other two had. We rejected it by conducting the “mean $\pm 1\sigma$ ” test, where the data for second trial fell out of the range of the test. This analysis was done in the excel.

Analyses by using peak height was performed. From the remaining 2 replicates, the mean value of signal at peak height was determined for each hydrocarbon. ‘F’ value was recognized as the ratio of relative detector response of the hydrocarbons, in order to solve for the composition of unknown mixture. In the analyses, F was set to be the relative detector response of signal with respect to the p-Xylene hydrocarbon. The ratio could also be set with respect to other things but to simplify things up, we stuck with only p-Xylene hydrocarbon.

After the ‘known mixture’ had been experimented, the same steps were repeated by using the unknown mixture. Unknown 2 was obtained from the lab instructor. Qualitatively analyzed, when the chromatogram for the second unknown was plotted, we determined that the **component of hydrocarbons present were Heptane, Cyclohexane and p-Xylene**. We figured it out by looking at the position of the peak height of the chromatogram, where it laid in the region of the pre-determined retention time of corresponding hydrocarbons, as shown in **Figure 6**. Quantitatively, by rearranging the equation of F, the mole ratio of components in the mixtures could be determined per trial, by referring to **Table 3**. The formula used were as listed in **Table 10**. Ultimately, the volume percent of each component in the unknown mixture was calculated. **p-Xylene, Heptane, and Cyclohexane’s volume percent are 35.37 ± 0.69 %, 29.73 ± 0.43 % and 34.91 ± 0.37 % respectively**. The values of the entire working were as shown in **Table 6 – 9** below.

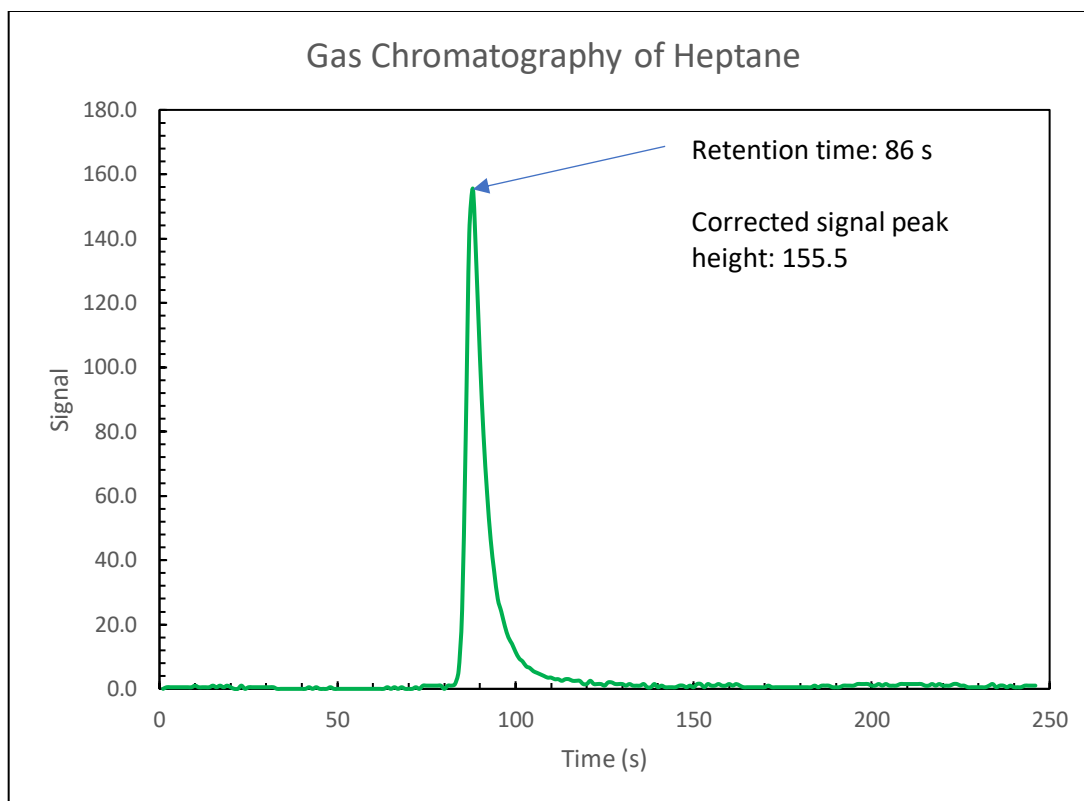


Figure 1: Gas Chromatography of Heptane

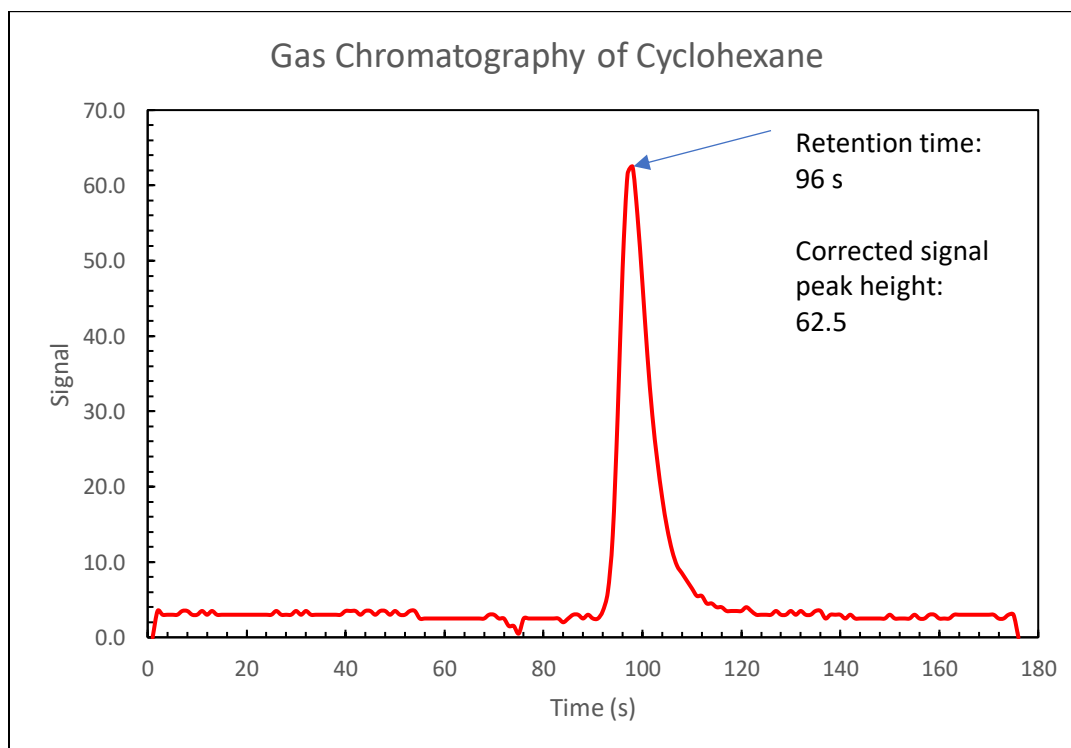


Figure 2: Gas Chromatography of Cyclohexane

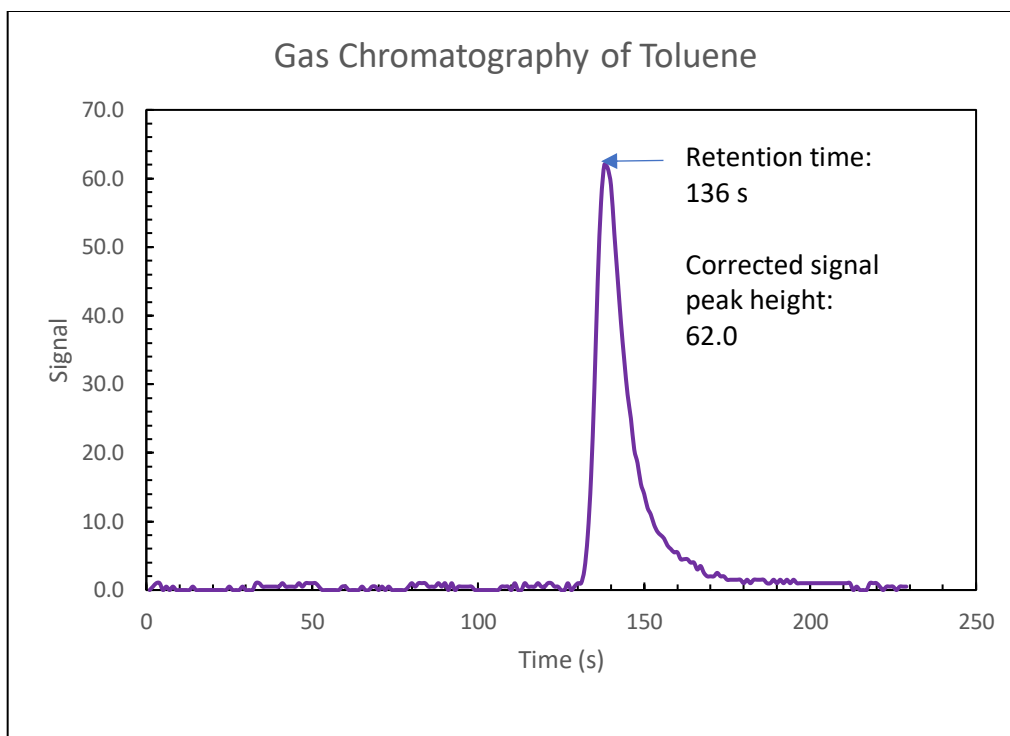


Figure 3: Gas Chromatography of Toluene

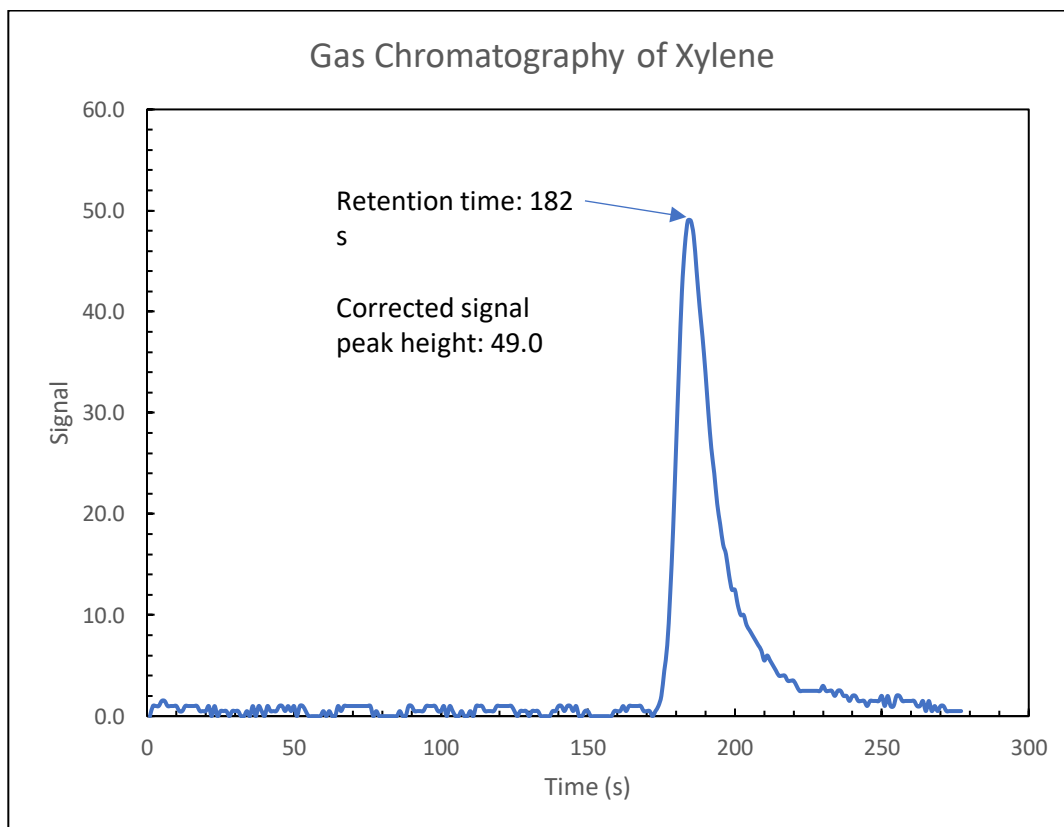


Figure 4: Gas Chromatography of Xylene

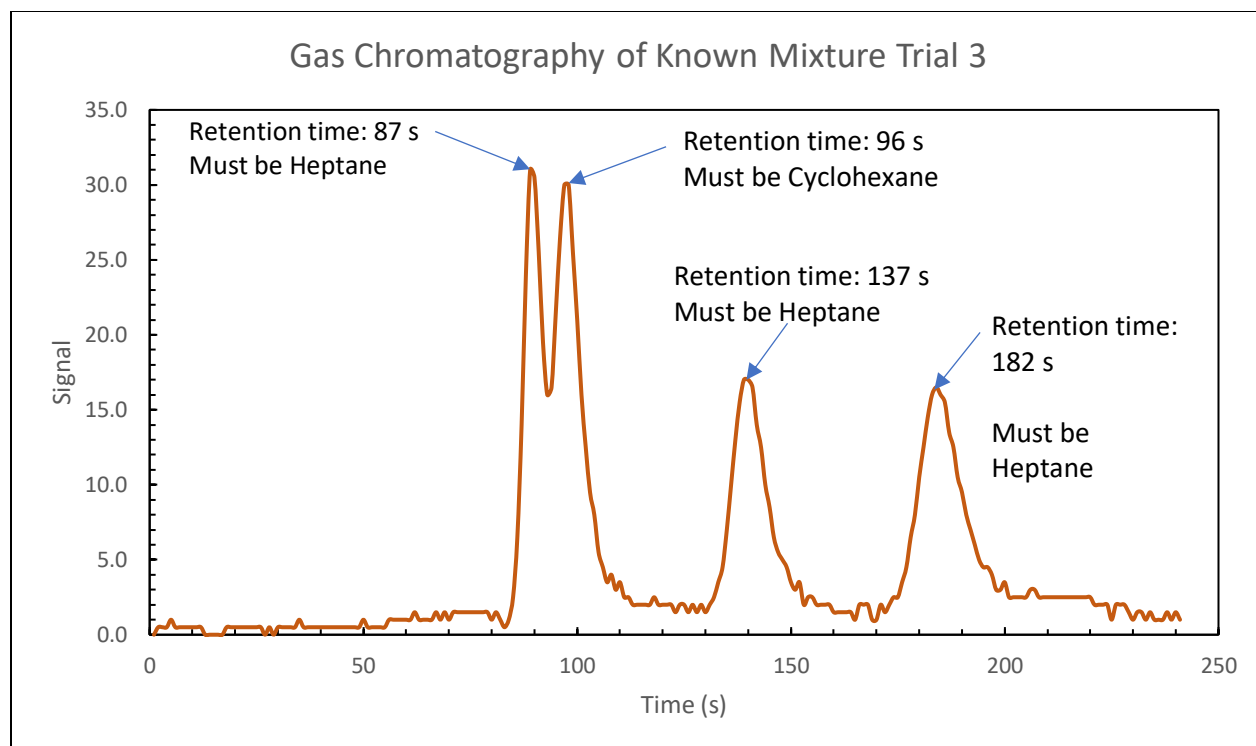


Figure 5: Gas Chromatography of Known Mixture Trial 3

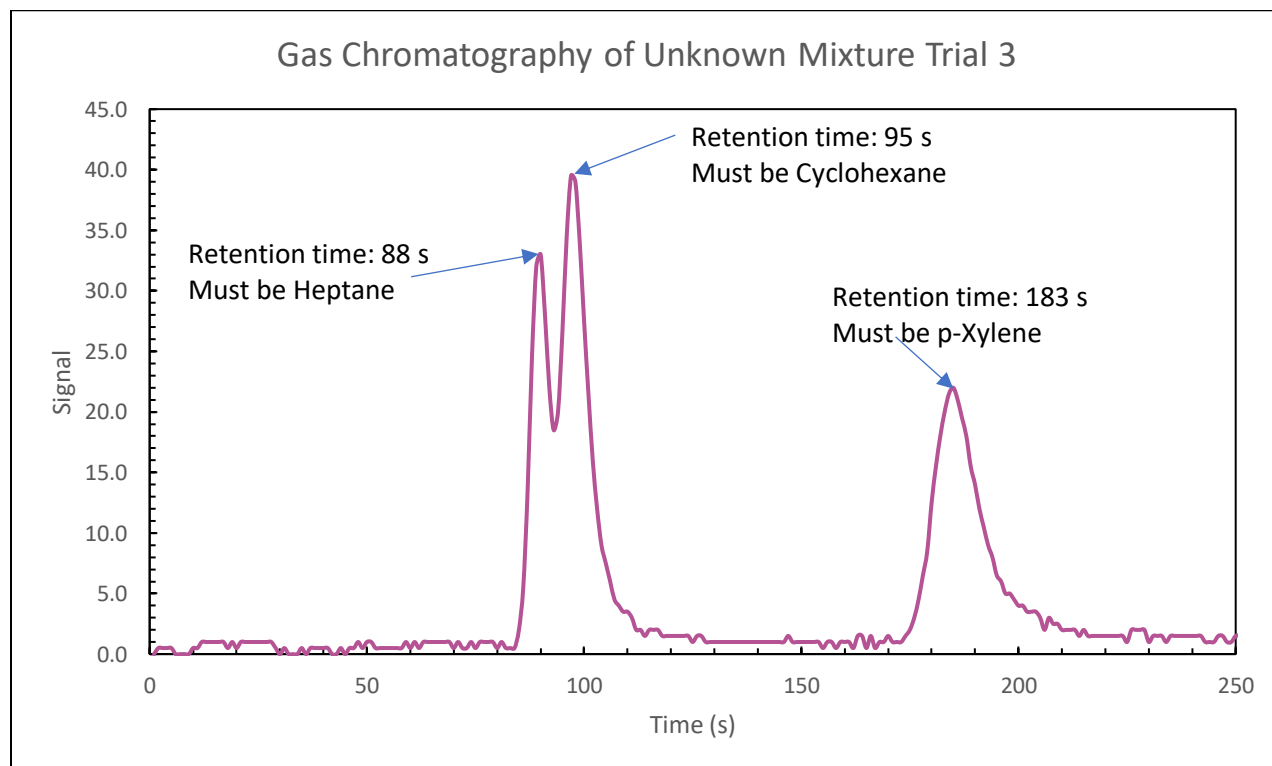


Figure 6: Gas Chromatography of Unknown mixture Trial 3

Component	Standard Peak Height				
	Replicate number		Average	Std. Dev	RSD (%)
	1.0	3.0			
Heptane	33.5	31.0	32.3	1.8	5.5
Cyclohexane	32.5	30.0	31.3	1.8	5.7
Toluene	16.5	17.0	16.8	0.4	2.1
p-Xylene	16.0	16.5	16.3	0.4	2.2

Table 1: Replicate measurement of Known mixture

Component	MW (g/mol)	Density (kg/m ³)	Mass (g)	Moles (mol)	Volume (m ³)	Component Percent (%)		
						Weight	Mole	Volume
Heptane	100.21	684	0.2049	0.002045	3.00E-07	24.17	22.95	27.95
Cyclohexane	84.16	779	0.2212	0.002628	2.84E-07	26.09	29.51	26.49
Toluene	92.14	867	0.1830	0.001986	2.11E-07	21.59	22.30	19.69
p-Xylene	106.16	861	0.2387	0.002248	2.77E-07	28.16	25.24	25.87
Total			0.8478	0.008908	1.07E-06	100.00	100.00	100.00

Table 2: Mass of each component in the known mixture

Standard Ratio		Signal		F with respect to xylene
Compound	Mole %	Mean	Std. Dev	
Heptane	22.95	32.25	1.77	2.18
Cyclohexane	29.51	31.25	1.77	1.65
Toluene	22.30	16.75	0.35	1.17
p-Xylene	25.24	16.25	0.35	1.00

Table 3: F value with respect to p-Xylene for each hydrocarbon

Component	Retention Time (s)
Heptane	86
Cyclohexane	96
Toluene	136
p-Xylene	182

Table 4: Retention time of each hydrocarbon

Component	Standard Peak Height					
	Replicate number			Average	Std. Dev	RSD (%)
	1	2	3			
Heptane	40.0	34.5	33.0	35.83	3.69	10.29
Cyclohexane	47.5	42.0	39.5	43.00	4.09	9.52
p-Xylene	25.0	22.5	22.0	23.17	1.61	6.94

Table 5: Replicate peak height for unknown mixture

Run 1		
Component	Peak Height	F with respect to Xylene
Heptane	40.0	2.182
Cyclohexane	47.5	1.645
p-Xylene	25.0	1.000

nX/nH		1.364	
nX/nC		0.866	
nX + nC + nH (mol)		1.000	
nX (mol)	0.346	42.69	Vol. of X (mL)
nH (mol)	0.254	37.19	Vol. of H (mL)
nC (mol)	0.400	43.20	Vol. of C (mL)
Total mole (mol)	1.000	123.09	Total Vol. (mL)

Table 6: Volume of each component in trial 1 of unknown mixture

Run 2		
Component	Peak Height	F with respect to Xylene
Heptane	34.5	2.182
Cyclohexane	42.0	1.645
p-Xylene	22.5	1.000

nX/nH		1.423	
nX/nC		0.881	
nX + nC + nH (mol)		1.000	
nX (mol)	0.352	43.46	Vol. of X (mL)
nH (mol)	0.248	36.28	Vol. of H (mL)
nC (mol)	0.400	43.20	Vol. of C (mL)
Total mole (mol)	1.000	122.94	Total Vol. (mL)

Table 7: Volume of each component in trial 2 of unknown mixture

Run 3		
Component	Peak Height	F with respect to Xylene
Heptane	33.0	2.182
Cyclohexane	39.5	1.645
p-Xylene	22.0	1.000

nX/nH		1.455	
nX/nC		0.916	
nX + nC + nH (mol)		1.000	
nX (mol)	0.360	44.37	Vol. of X (mL)
nH (mol)	0.247	36.24	Vol. of H (mL)
nC (mol)	0.393	42.43	Vol. of C (mL)
Total mole (mol)	1.000	123.04	Total Vol. (mL)

Table 8: Volume of each component in trial 1 of unknown mixture

Volume percent (%)					
Compound	Run 1	Run 2	Run 3	Mean	Std. Dev.
p-Xylene	34.69	35.35	36.06	35.37	0.69
Heptane	30.22	29.51	29.45	29.73	0.43
Cyclohexane	35.10	35.14	34.49	34.91	0.37

Table 9: Volume percent of each component in the unknown mixture

Formula	
Moles (mol)	Mass (g) / Molar mass (g/mol)
Weight percent (%)	Weight of compound (g) / Total weight of compound (g) * 100 %
Mole percent (%)	Mole of compound (mol) / Total moles of compound (mol) * 100 %
Volume percent (%)	Volume of compound (m ³) / Total volume of compound (m ³) * 100 %
F with respect to p-Xylene	Signal per mole of compound / Signal per mole of p-Xylene
Average height	AVERAGE(values) function in excell
Standard deviation	STDEV(values) function in excel
RSD (%)	Standard deviation / Average
Volume of compound (mL)	Moles of compound * Molecular weight * 1000 / Density
<p>$F = S_n / S_x$; S_n is signal per mole of compound n, S_x is signal per mole of p-Xylene.</p> <p>$S_n = H_n / n_n$, where H_n is the peak height and n_n is the moles of compound n.</p> <p>By rearranging the equation of F, we can express $n_x/n_n = F_n * H_x/H_n$.</p>	
<p>F_n will be the same in both known and unknown mixture, hence we can just solve simultaneously for the mol ratio since we have 3 equations with 3 unknowns</p>	

Table 10: Formula used to calculate the values tabulated in all tables

Compound	Retention Time (s)	Width	Resolution with respect to nearest peak
Heptane	88	12	0.333
Cyclohexane	95	30	0.333
Xylene	183	52	2.146

Table 11: Resolution of each compound of the unknown mixture

Based on **Table 11**, the resolution of each compound was calculated. The resolution was determined by comparing the desired peak to the nearest peak. These data were obtained from examining the chromatogram in Figure 3. The resolution of heptane and cyclohexane were determined with respect to each other, while xylene's resolution was determined with respect to cyclohexane, since that was the closest peak to it. The resolution measured how well 2 elution peaks could be differentiated from each other in the chromatographic. The bigger the resolution value, the easier to differentiate the peaks in between two compounds.

My analyses only encompassed the peak height of the chromatogram. By comparing the results that I obtained with one of my teammates who did the analyses based on peak area, she (Linh Nguyen) managed to have smaller volume percent for **Heptane** ($23.84 \pm 2.17 \%$) and **Cyclohexane** ($30.83 \pm 1.10 \%$) but with bigger standard deviations, and a bigger volume percent and standard deviation for **p-Xylene** ($45.33 \pm 1.35 \%$). Theoretically, analyzing by using the peak area is more accurate in comparison to the peak height. However, based on these results, it is obtained that the standard deviations of peak areas for each component of the mixture are greater than the standard deviations of peak height. This may be because of error in calculations when computing the area, since the approach seems to be more complicated than the peak height. There seems to have a significant amount of standard deviation of the data collected. This may be because of the error made during the experiment, which includes the mishandling of volatile compounds. When inserting the volatile hydrocarbons into the GC machine, it should be injected and removed immediately. A delay in doing so will cause some portion of the compound to vaporize since it is highly volatile. This affect the amount of each component that actually exist in the unknown mixture.