Gas Chromatography.

Student's name.

Institution Affiliation.

Course code.

Date.

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Introduction

Gas chromatography (GC) is an effective separation unique technique which analyses mixtures of volatile substances. It is quite simple in principle and helps in identifying the components of volatile compounds as well as the quantity of each component. This method can also be used in determining the purity of a certain mixture and performing qualitative analysis on the mixture. The most popular used procedure considers chemical analysis. It also was one of the first analytical machine associated with a computer which can observe the analysis, process the data and state the results (Scott, 1997). In this experiment, the Agilent 6850 series GC was used which is temperature programmable fitted with FID detector and 0.32mm capillary column, polydimethylsiloxane stationary phase, 15m long.

Figure 1 below illustrates the schematic description of gas chromatography. The instrumentation of the entire process is quite simple. The central item in the apparatus that are featured below is the column which is made up of a long narrow tube that is permeably packed with an absorbent. The most common technique used at this point is the elution method where a mixture of chemically inert gas known as the carrier gas passes continuously through the column. At the same time, the mixture that is to be separated is also passed abruptly through the inlet of the column. After the injection, the mixture is then swept by the carrier gas into the inside of the column. The components of the mixture are then transported through the column at different rates based on the nature of each component.

Therefore, these components will then be separated into different zones and emerge from the column at different times where they are detected. The common system usually uses a flow rate controller. Number of moles of the carrier gas through the controller per unit time is continuous. Sampling system allows the injection in this steam of gas. This sample is then

vaporized in a short term of time and inserted into the column. The column included in temperature picked out usually lies in the range ambient temperature to 350 C. A detector transfers a signal faction of the composition of the carrier gas which states the identification of each component after the components of mixture are injected at the column and pushed downstream by the carrier gas (Guiochon & Guillemin, 1988).

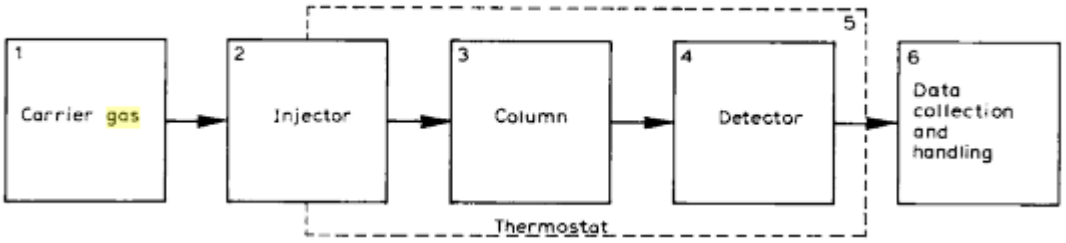


Figure 1: Schematic description of gas chromatography

There are two kinds of columns commonly used in gas chromatography which are packed columns and capillary columns. Packed columns have an internal diameter of 2 to 3 mm and typically 2 to 3 m long. The packing consist of a solid support such as alumina or carbon where the size of the particles ranges from 5 to 150 mm. On the other hand, the capillary columns have an internal diameter of 0.5 to 0.15 mm and they are generally 12 to 60 m long. They are made up of fused silica tubing that is coated on the outside with a polyamide that gives the tubing strength. The most commonly used injector for capillary columns is the heated split/ split less injector. However, the capillaries, in this case, take less amount of sample as compared to the packed columns.

There are a number of advantages derived from using the split injection mode. One is that a small number of parameters for optimization and the other is that shorter time is required for analysis because there is no need for a low initial column temperature. The technique is good for thermolabile sample due to the short residence time in the hot injector (Sparkman, 2011, P.52). Another key concept in this process is the retention time. It refers to

the distance between the injection point and the centre of a peak along the axis of the chromatogram. Generally, this is the time taken before a certain zone in a mixture transverses the column. The qualitative analysis of an unknown mixture is determined through comparison with chromatogram that is obtained from substances that are well known.

Quantitation by GC is very popular analytical technique. The significant benefit of GC is their wide dynamic range. The detectors can perform measurements in large quantities and very small quantities. There are two common problems with GC, one is that it injects too much sample. Overweight usually gives poor integration values and cause peak asymmetry. Another problem is that split injectors do not always provide reproducible splits resulting in poor external standard calibrations. The preferred method of quantitation is the internal standard method which has two advantages. One is the independence of the injection site. And the detector response is linear. This experiment is designed to know GC operation with various critical parameters such as oven temperature, flow rate as well as injection time.

Method

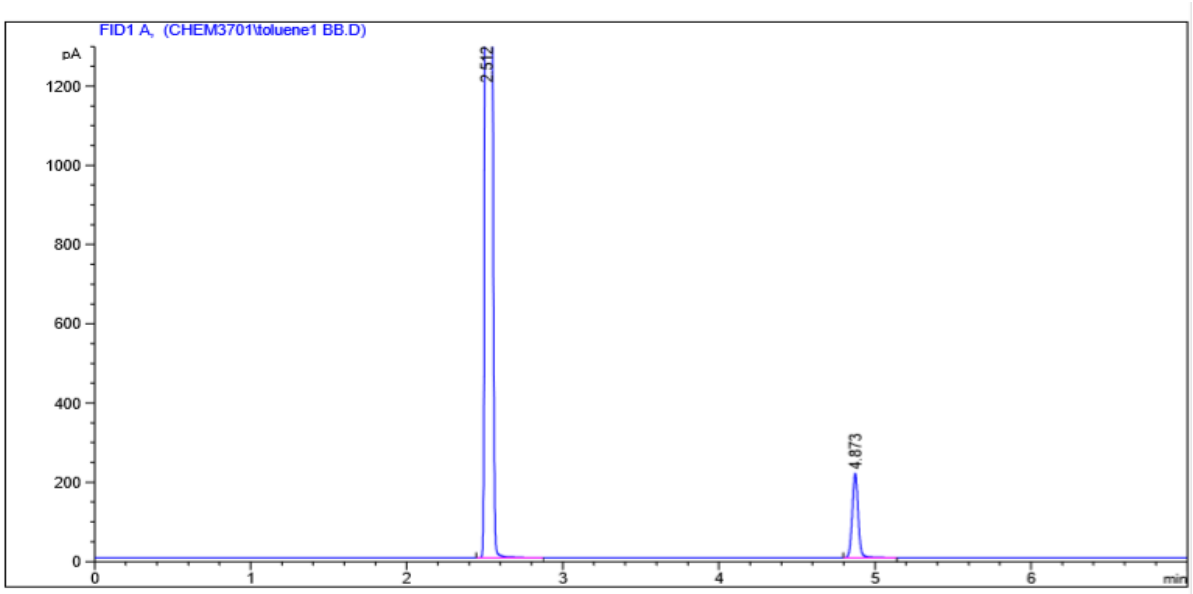
The sample of toluene (0.05 g) was papered several times in dichloromethane. First with an oven temperature of 50 C by using 1A method. Second the same sample was run using 1B method with an oven temperature of 70 C. And the third time was with a column flow of 0.9 ml/min using method 1C and finally testing injection size by using (5 ml) of toluene in dichloromethane with 1 A method.

After that, 10% v/v from crude oil solution was run ten minutes with oven temperature 140 C and column flow 1.3 ml/min using method 2A. The injection was repeated with an oven temperature of 60 C using 2B method.

Also, 10 drops from crude oil solution with 10 drops toluene solution were mixed using the 2B method. Ethyl benzoate (1.0) was added to 1.0 ml of the crude oil solution. The

sample was injected using 2B method and run about 6.8 minutes. 0.10 g ethyl benzoate was added to the toluene solution and run through the GC

Result



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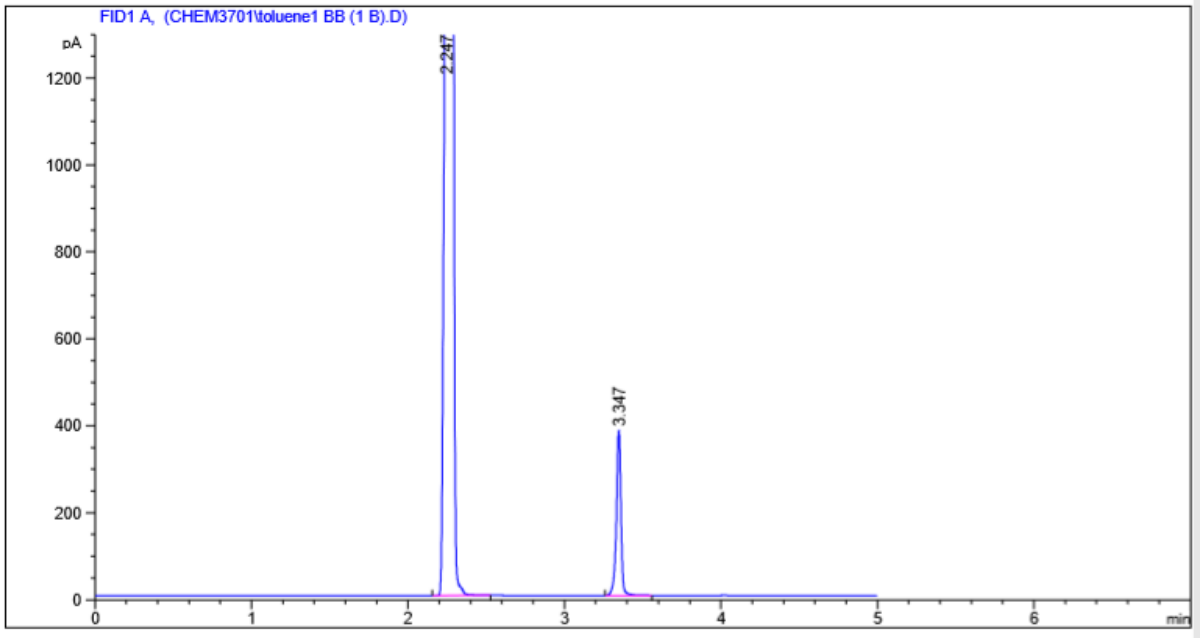

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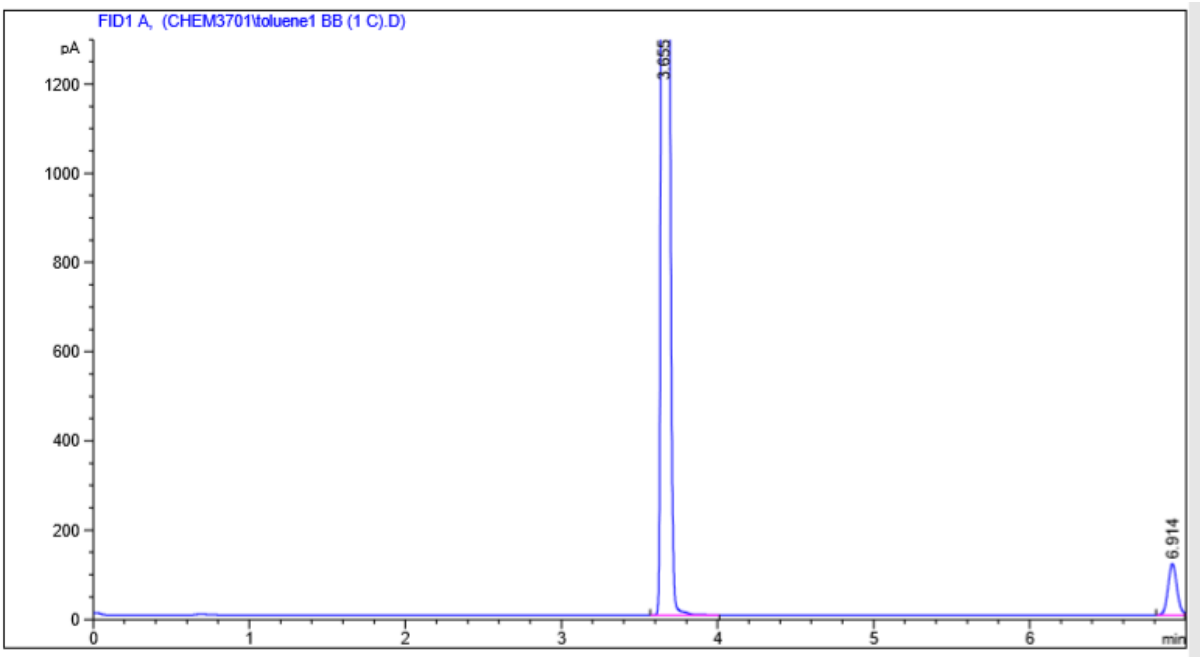

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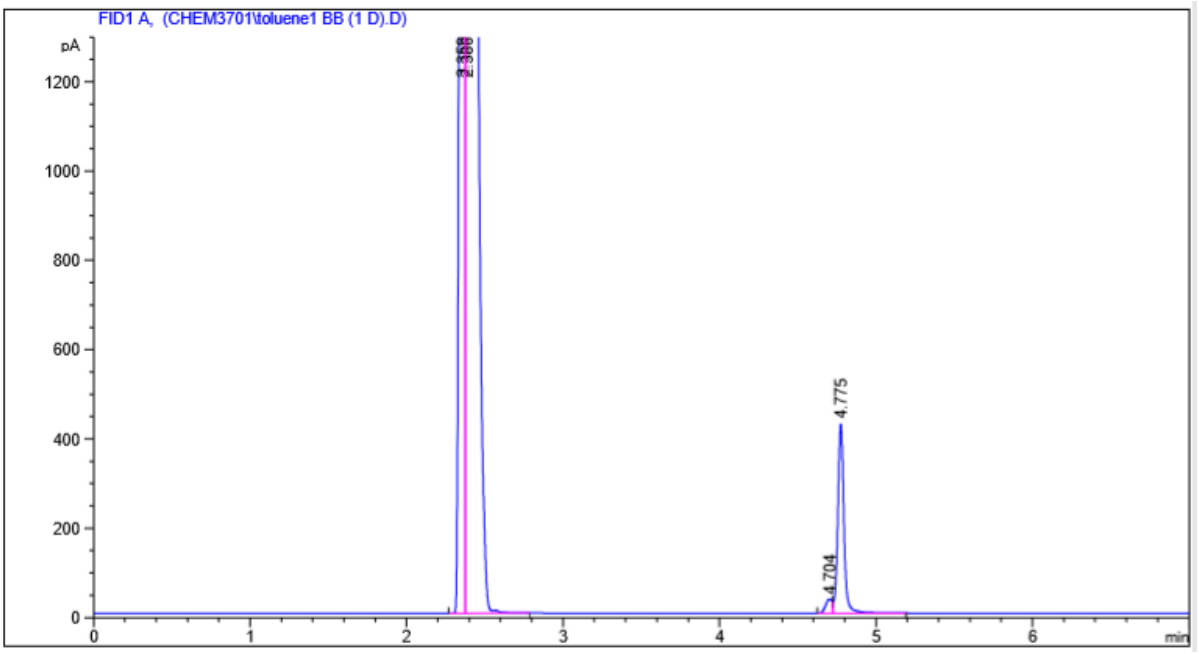

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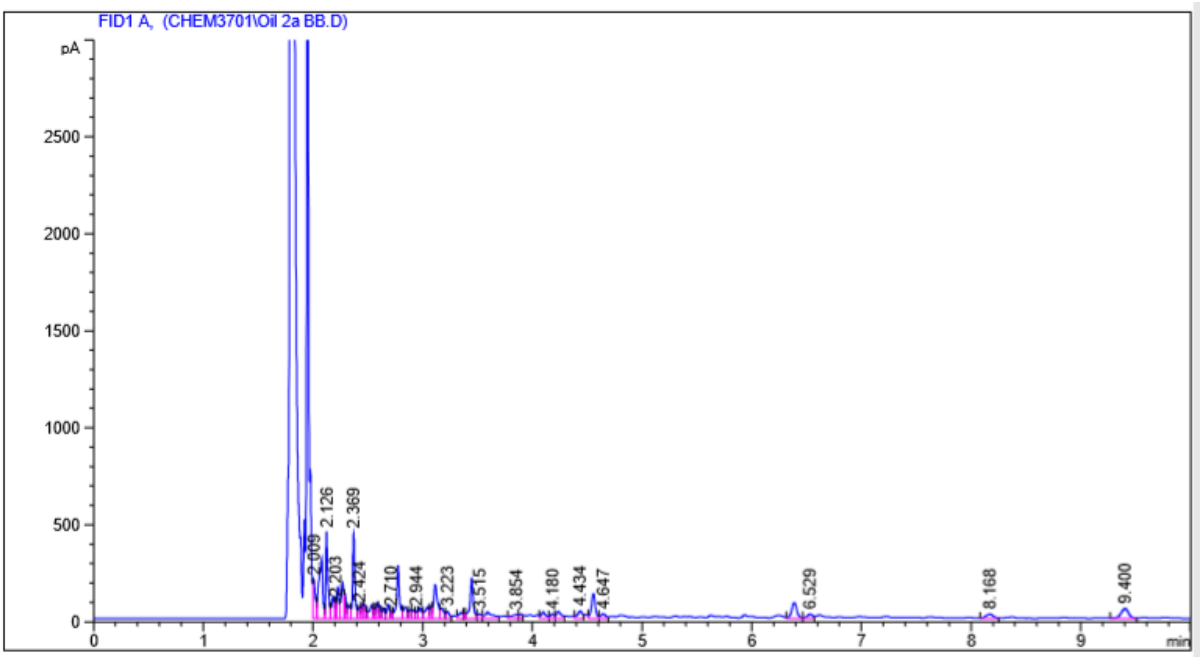

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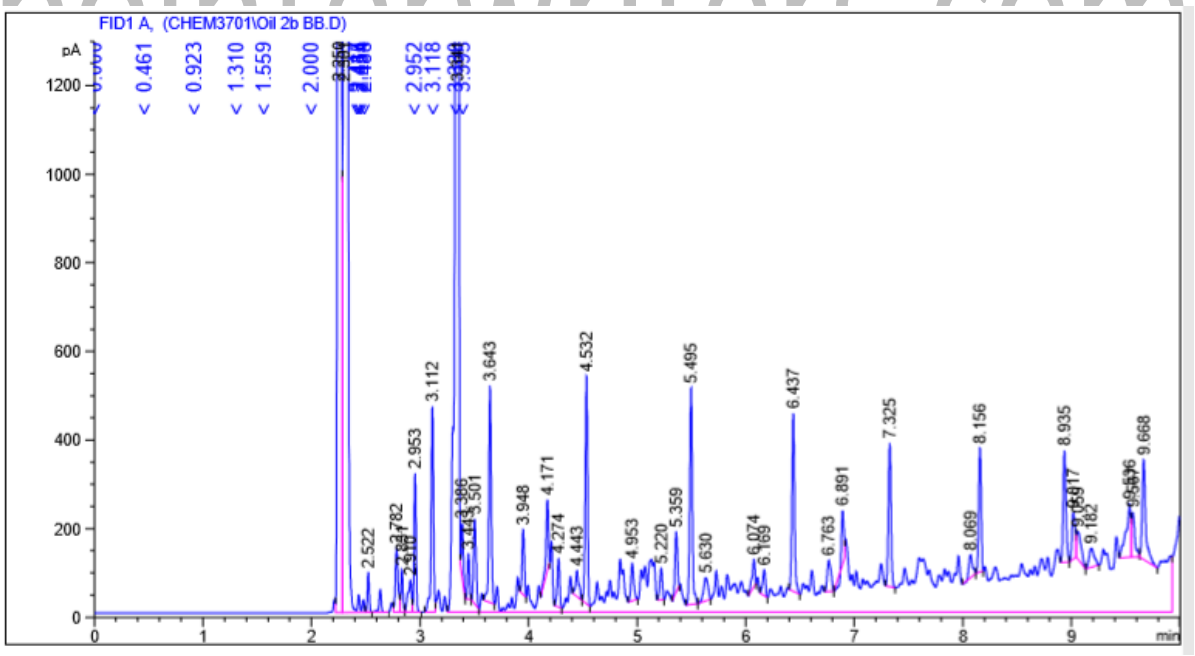

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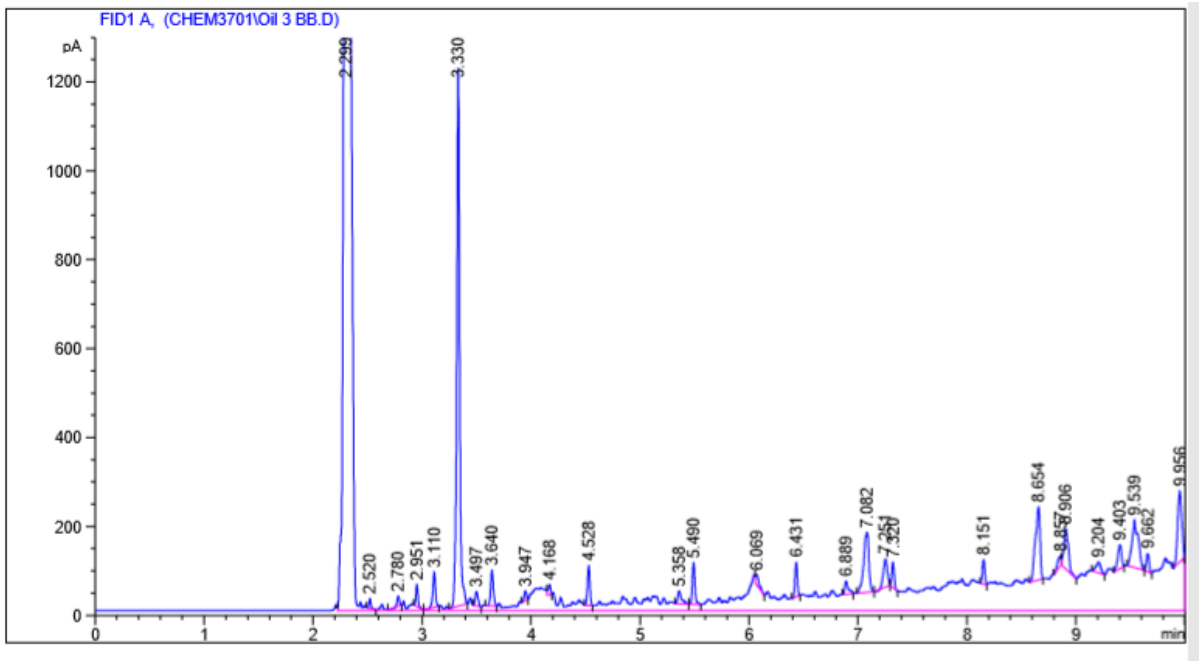

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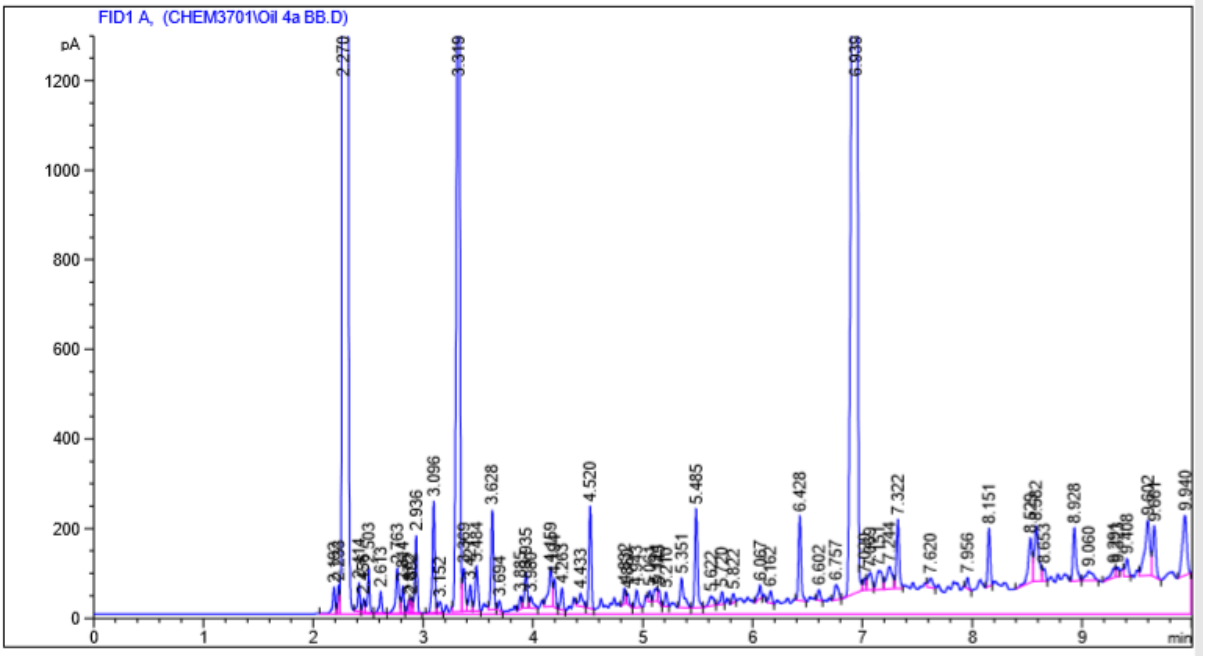

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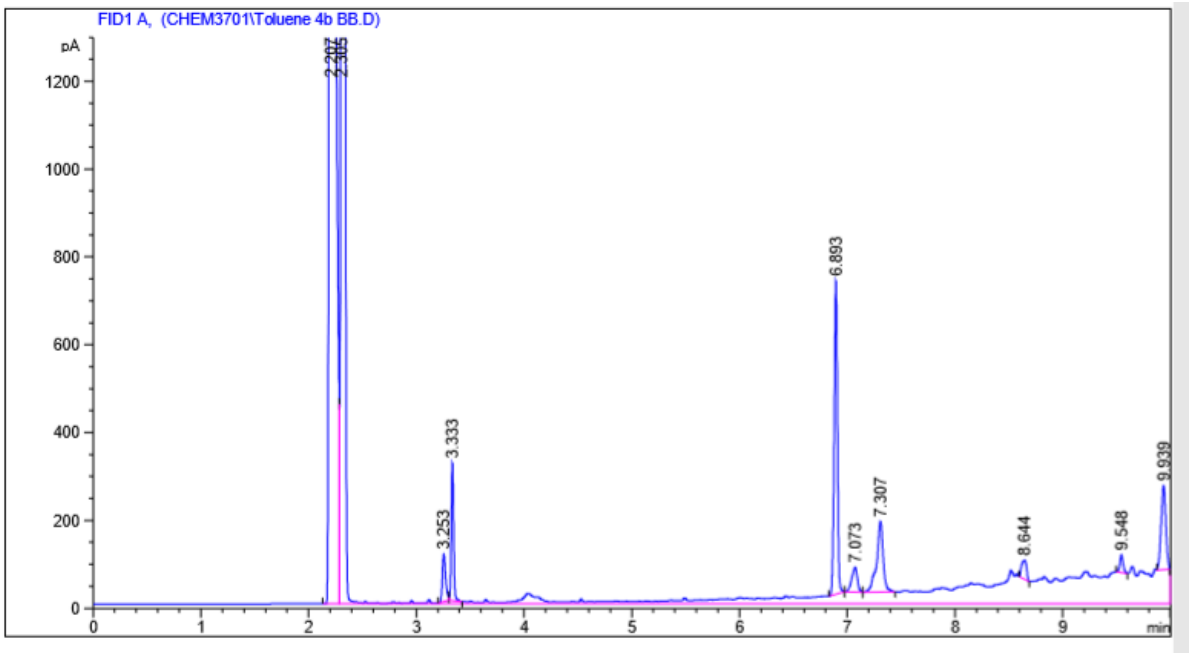

Discussion

The sample was injected and vaporized as well as pushed through the column by inert gas to facilitate separation. As shown in figure 1 the result showed the sample of toluene 0.05 g in dichloromethane at 60 oven temperature and 1.3ml/min column flow. The retention time of toluene and dichloromethane measures the distance between 0 and toluene peak which is 2.5 mins and 4.87 mins for dichloromethane. The concentration of the sample is $\frac{0.05}{0.025}$.

When the temperature increased to 70 C in figure 2, the distance will get smaller and this effect the separation. Overall, high temperature affords poor separation. The size of the peak as compared to the first injection was the same because the concentration was also the same. The shape at 1b is slightly wider in high temperature. The retention time remained almost the same for the toluene peak when the temperature raised while it decreased to 3.34 at dichloromethane. In figure 3, the first step is repeated, and the column flow was changed to 0.9ml/min using the 1C method, the column flow was decreased in this figure. The

retention time gets higher at 1c (3.6) at toluene peak and 6.91 for dichloromethane peak compared with the retention time at 1a which lead to better separation.

The difference between peaks in 1a= 4.873-2.512= 2.361

The difference between peaks in 1c = 6.914-3.655= 3.259

As a result, decreased flow rate gives great separation.

Figure 4 shows the sample examined by injection size which contains 1.3 ml/min flow rate and 50 C temperature with 5mL concentration. The peaks are significantly wider. Consequently, poorer resolution. Also, Separation= 4.775-2.358= 2.417 compared with the difference between peaks in 1a =2.361 which is almost similar. The retention time at 1d is 2.358 and 2.417 for the two peaks shown and for 1a is similar which were 2.512 and 4.873. When the concentration increased the resolution get decreased and the peaks will be getting wider. It is unreliable when the detector is saturated as demonstrated by a flat top peak.

Figure 5 shows the effect of the temperature by injecting 10% v/v crude oil solution at 140 C oven temperature with 1.3 ml/min flow rate. As shown there are about 17 peaks detected from 1.5 – 9.4 min. High temperature gives poor separation. Signals are sharp which means well resolved. Figure 6 display result from the same injection using 2b at 60 C. Improvement of resolution is remarkable. The number of peaks significantly increase. The signal intensity = 6. In addition, the first peak in figure 7 refer to toluene (2.5) and this peak stays the same when adding 10 drops of crude oil mixed with 10 drops toluene in figure 8. The other peaks from crude oil were decreased.

The concentration of toluene in crude oil =

$$\frac{A_x}{C_x} = F \times \frac{A_s}{C_s}$$

$$c_s = 7 \times 10^{-3}\ M$$

$$A_s = 9705.87$$

F= 0.38

$$A_x = 6.06x1O^4$$

$$\frac{6.06x1O^4}{c_x} = 0.38\ x\ \frac{9705.88}{7x \cdot 10^{-3}}$$

$$\frac{6.06x1O^4}{c_x} = \frac{1}{0.38} \times \frac{9705.88}{7x \cdot 10^{-3}}$$

$$c_x = \frac{6.06x1O^4}{0.38} \times \frac{9705.88}{7x \cdot 10^{-3}}$$

$$c_x = 158157.8\ \times\ 721 \times 10^{-7}$$

$$c_x = 0.114\ M$$

Conclusion

This experiment shows several runs with toluene in dichloromethane at several instrument settings as well as qualitative and quantitative analysis of crude oil. Several critical parameters including oven temperature, flow rate, and injection size were also observed.

References

The manual for CHEM3701

Guiochon, G., & Guillemin, C. L. (1988). *Quantitative gas chromatography for laboratory analyses and on-line process control* (Vol. 42): Elsevier.

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