**INSTRUMENTAL ANALYSIS LABORATORY**

**NAME OF THE EXPERIMENT:** GC

**DATE OF THE EXPERIMENT:** 15.05.2023 – 16.05.2023

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**GROUP NUMBER:** 6

**DATE SUBMISSION:** 21.05.2023

**INSTRUMENTATION**

Carrier Gas Tank

|  |
| --- |
|  |

Column

Flow Meter

Sample

Display

Data System

DETECTOR

A close-up of a spiral

Description automatically generated with low confidence

Sample Injection

Chamber

OVEN

Flow Regulators

Syringe

Dry Air Tube

H2(g)

Tube

THERMOSTAT

**The brand of gas chromatography device is GC-2010 Shimadzu.**

**Carrier Gas Tank**: The mobile phase of gas chromatography is carrier gas. Its chemically desirable property is that it is inert. The most suitable gas for this mobile phase is He. Besides, Ar, N, H can also be preferred in terms of usage. The tank, is the place where the mobile phase is kept.

**Flow Regulators:** It is used to control the gas flow rate in the carrier gas system and is required for GC.

**Sample Injection Chamber:** The slow addition of the sample to the system, or not being in the desired dimensions for GC reduces the column efficiency and solubility. In GLC, the sample is in liquid form. The sample, injection chamber is, inside an oven in the GC, so the sample becomes gaseous. The desired sample size for capillary columns is 100 times or smaller than, 20 µL, so a sample splitter should be used. The sample is transmitted to the column with this split, and the rest is sent to waste. The split injection method was also used in this experiment**.**

**Syringe**: A micro syringe, Hamilton Boraduz Schwaz Microliter #701, was used to load the sample into the sample injection chamber**.** The tip of the syringe must be sharp because the septum must be punctured for sample injection.

**Column:**In GC, the column, is the stationary phase. ,The columns can be packed or capillary. The column used in the experiment is the open tubular capillary column. It is 30 meters long, and, in the column, a viscous polymer (5%-phenyl)-methylpolysiloxane),, a non-polar liquid form, is used. Being an open tubular column increases efficiency,. The diameter of the column is 0.53 mm, and the film thickness is 5 μm.

**Detector:** The detector used in the experiment, Agilent Technologies, Inc. 19095J-623, is a branded flame ionization detector. It has a mass-sensitive feature. ,The flame in the detector is obtained by igniting dry air and hydrogen gas, and the gas sources are outside the GC in gas tubes. It is preferred for organic molecules.

**Display:** The data from the detector is collected, and the reading is converted to be displayed ,in the software., Thus, qualitative analyzes can be made from GC with the obtained chromatograms. The software in which the data is read belongs to Shimadzu.

**CALCULATIONS**

**1)**

**Table 1.** At 40℃ Retention Times for Standards

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **MeOH** | **IPA** | **DCM** | **n-Hexane** | **n-Butanol** |
| 0,823 | 1,411 | 1,547 | 2,560 | 5,153 |

There are In MeOH, standards and unknown solution.Therefore, all observed chromatograms contain MeOH, but only the first data is noted.

|  |  |  |  |
| --- | --- | --- | --- |
| **Mix 1,** |  |  |  |
| tR,1 | tR,2 | tR,3 |  |
| 1,576 | 2,514 | 4,745 |  |
|  |  |  |  |
| **Mix 2,** |  |  |  |
| tR,1 | tR,2 | tR,3 | tR,4 |
| 1,555 | 2,487 | 4,674 | 1,346 |

**For Mix 1,**

**Table 2**. Peaks, Retention Times, and Closeness to Standards

|  |  |  |
| --- | --- | --- |
| **Peak** | **Retention times** | **Closeness to Standards** |
| First | 1,576 | DCM |
| Second | 2,514 | n-Hexane |
| Third | 4,745 | n-Butanol |

When the data in Table 2 is examined, it is observed that mixture 1 contains DCM,n-Hexane and n-Butanol.

**For Mix 2,**

**Table 3**. Peaks, Retention Times, and Closeness to Standards

|  |  |  |
| --- | --- | --- |
| **Peak** | **Retention times** | **Closness to Standards** |
| First | 1,346 | IPA |
| Second | 1,555 | DCM |
| Third | 2,487 | n-Hexane |
| Fourth | 4,674 | n-Butanol |

When the data in Table 3 is examined, it is observed that mixture 2 contains IPA,DCM,n-Hexane and n-Butanol.

**2)**

**Table 4**. Retention Time Values for Mix 2 at Different Temperatures

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Temperature,°C** | **DCM,** | **Hexane,** | **IPA,** | **n-Butanol,** |
| 40 | 1,555 | 1,346 | 1,346 | 4,674 |
| 60 | 1,127 | 1,601 | 0,951 | 2,491 |
| 80 | 0,872 | 1,136 | 0,787 | 1,596 |
| 100 | 0,745 | 0,903 | 0,691 | 1,16 |
| 120 | 0,685 | 0,787 | No Data | 0,973 |

**Graph 1. Mix 2 Retention times of Peaks at Different Temperatures**

**3)**

**Table 5.** At Different Temperatures and Temperature programs values of n-Butanol widths for Mix 2

|  |  |
| --- | --- |
| **Temperature (℃)** | **Width(w) of n-Butanol (min)** |
| 40 | 0,570 |
| 60 | 0,303 |
| 80 | 0,270 |
| 100 | 0,149 |
| 120 | 0,165 |
| Temperature Programing | 0,298 |

**Calculation of Widths(W):** W = tR,f  -tR,i

tR,f  = Final retention time tR,I = initial retention time

N: Number of theoretical Plates H: Height of plate L:Column length :30 m

*H = L/N*

**At 40℃,** **H**=(3000cm/1075,8) = 2,789 cm

For other temperatures following by that calculation,

**Table 6.** The theoretical plates and plate heights of n-Butanol values for Mix 2 at 30°C-120 °C

|  |  |  |
| --- | --- | --- |
| **Temperature (℃)** | **N** | **H(cm)** |
| 40 | 1075,8 | 2,789 |
| 60 | 1081,4 | 2,774 |
| 80 | 559,1 | 5,366 |
| 100 | 969,8 | 3,094 |
| 120 | 556,4 | 5,392 |
| Temperature Programing | 2082,8 | 1,44 |

**QUESTIONS**

1. The column, detector and sample injection chamber are the heated sections in the GC.
2. The sample's polarity and temperature affect the samples' retention times in GC. As can, be seen because of the operations performed in the Calculations part, the time to reach the detector is prolonged for the samples if temperatures decrease. The gas flow rate, the column length, and the polarity may also have changed over the retention time. The boiling point for the sample is affected positively by the polarity of the sample. The reason polarized samples reach the detector faster than other nonpolar molecules, is that the column used in this experiment is nonpolar. The speed of acquiring the detector for the samples cannot be determined by simply comparing the boiling points of the samples. The retention times of IPA, DCM, n-Hexane, and n-Butanol were investigated, in the experiment, and the retention times increased from IPA to n-Butanol. Towards n-hexane, DCM, IPA, and n-Butanol, the polarity of the samples increases, and since n-Butanol is the most polar sample, it is expected to have the highest boiling point. The time the molecules need to reach the detector cannot be determined by looking only at the boiling points of these molecules because the polarity of the samples also affects this time. As proof of this, the polarity of DCM is IPA' and retention time for DCM is less than for IPA. However, although n-Hexane is the minor polar compound among the samples, its retention time is longer than the retention, time of DCM and IPA and shorter than the retention time of the n-butanol. The column temperature affects the time required for the sample to reach the detector. If the temperature is increased, the, retention time is expected to decrease. As proof of this theory, it was observed that the samples' retention times decreased with the temperature increase in the second part of the experiment. A decrease in resolution is observed in case the peaks overlap or intertwine at high temperatures. ,An example is the inability to obtain data for IPA at 120℃.
3. The time of the samples to reach the detector is affected by the column temperature. The retention time is expected to decrease with the temperature increase. As proof of this, theory, in the second experiment, retention times decreased from 40°C to 120°C for hexane butanol and DCM, with increasing temperature. At 120 °C, this time decreased, and data could not be obtained due to resolution reduction.
4. In the experiment, the temperature programming was set to be in the range of 30°C-120°C. Low resolution and fast access to the detector are observed at high temperatures, and for DCM, IPA, and n-hexane,, retention times increased with temperature programming. On the other hand, n-Butanol' decreased this time.

**5)** It is possible to improve the observation of retention times due to the sherpening of the peaks with increasing column efficeincy. Therefore, temperature programming should be preferred for dividing the mixture into two wells.

1. The molecular weight of the samples affects the peak areas observed in the chromatogram. The number of ions in the flame provided by the flame ionization detector is close to the number of ions in the carbons. As a result of this situation, the flame ionization detector used becomes easier to detect carbon atoms. Also, the flame ionization detector has a mass-sensitive feature. As a result of this feature, it is observed, that the peak areas observed in the chromatogram increase as the molecular weights of the samples increase.
2. Setting a temperature above the room temperature, which is adjusted to be above the boiling points of all samples. This temperature adjustment is because the samples are in the liquid state at room temperature; however, the samples must be in the gas state to perform the separation process in gas chromatography.
3. Dilution of samples is necessary for systems using capillary columns in gas chromatography because the samples in the columns used are smaller than those in regular packed analytical columns. Therefore, the sample splitter is used to dilute the samples. These splits send all the analytes to the column and the rest to the waste section.
4. The reasons for this may be that different samples have the same or very close boiling points or have very similar polar properties. ,On the other hand, if the length of the columns used in the system is shorter than it should be, it causes the retention times to be similar for different samples and effects the completeness of the separation. If there is a flow rate faster than it should be, this may lead to similar retention times that affect the seperation negatively. High temperatures may also reduce the resolution and prevent a good and precise separation from being observed.

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