Lab 8

Gas Chromatography

11/5/2019

Objective

The purpose of this experiment is to determine the composition of hydrocarbon mixtures unknown prepared by the TA by using gas chromatography method.

Introduction

4 standard compounds : heptane, cyclohexane, toluene, & p-xylene, are used in this gas chromatography method. The retention time (time required to pass through a column in the chromatography) for each compound is determined, in order to qualify & quantify the given unknown. The composition of the unknowns is determined before quantify each component in the unknown, by comparing the retention time obtained from the unknown with the standard compounds' values.

Procedures

A) GC Operation & Injection Procedure

1) Take notes the proper technique for injecting samples with the microliter syringe, and the proper procedure to set up the data logger.

2) Make notes of the values on the knobs of the machine. Do not change the knobs.

B) Determination of Retention Times, t_r.

3) Investigate known compounds. Small samples of the 4 pure hydrocarbons will be provided in separate sealed vials.

4) Separately inject ~ 1.0 uL of the pure hydrocarbons & obtain 4 chromatograms from which the t_r of each component can be measured.

Lab 8

Gas Chromatography

11/5/2019

5) Record the retention times in seconds. This can be read from the chart by measuring the distance from injection to the peak maximum & dividing by the chart drive rate (mm/second).

6) 1 replicat is needed. Record the data on both the chart recorder & data logger. Transfer to USB drive.

C) Determining the components of Unknown

7) Qualitative analysis of an unknown mixture. Unknown sample is provided in a small vial. Required only one unknown. Record the label. Inject exactly 1.0 µL of unknown mixture. Make sure the temperatures & flow rate have not changed since injection. Identify the components in the unknown by comparison of the retention times of the peaks in the unknown with the retention times obtained with the standards. 1 replicate only is needed. Record the data.

D) Quantifying the components of Unknown.

8) Preparation & analysis of quantitative standard. Use gravimetry to prepare a 4-component mixture of H, T, C, & X for which the exact amounts of each is known. Determine the relative response factor Fi for each compound i. Obtain 3 or more replicate 1.0 µL elutions of the 4-compound standard mixture. Record the data on both the chart recorder & data logger.

64

Lab 8
Gas Chromatography
11/5/2019

- Unknown 2 used.

Known Mixture

H → 0.2049 g
C → 0.2212 g
T → 0.1830 g
X → 0.2387 g

Lab 8

Gas Chromatography

11/5/2019

6. Quantitative Analysis of Unknown Mixture. Make at least 3 replicates 1.0 µL-injections of unknown. Record the data on both chart recorder & data logger.