**DETERMINING THE IDENTITY AND PERCENT COMPOSITION OF A MIXTURE**

 **OF ALCOHOLS USING A GAS CHROMATOGRAPH**

INTRODUCTION

Gas chromatography is a technique used to analyze mixtures. The instrument allows mixtures to be separated and the amount of each component to be determined. One advantage of this technique is that very small (a few microliters) samples can be analyzed.

Samples to be analyzed in a gas chromatograph must be volatile, that is, they must vaporize easily. Once vaporized, the sample is carried through a long tube containing a porous material. A nonreactive gas such as helium is used to carry the components of the mixture through the tube which is usually referred to as the column. Not all components of a mixture travel through the tube at the same rate. Thus, some components will arrive at the detector at the end of the tube before others. The detector in this GC is called a thermal conductivity detector. As the components pass over the detector, the detector sends a signal to a recorder and a plot (chromatogram) is produced.

Using the chromatogram, the identity and the percent composition of each component in the mixture can be determined.

**The identity of the alcohols will be determined by using the retention time method.**

 \*To determine the retention time of each alcohol you will first measure the distance from

 the middle of the air peak to the middle of each peak.

 \*You will then need to convert the distances to a time in order to determine the retention

 time (Rt). The paper will be moving at a speed of 2 cm/min. Use this value and the

 distance you measured to set up a proportion to solve for the number of minutes it took

 each sample to travel through the column.

**The percent composition is directly related to the area of each peak in the chromatogram. The area of each peak can be found by using the following method:**

 \*The peak can be treated as a triangle and the formula, **Area = ½ Base x Height** can be

 used to determine the area of the peak.

 \*The percent composition for each peak is calculated by: adding together the peak areas

 to get the total area of the peaks, and then dividing each area by the total area and

 multiplying by 100.

PURPOSE

To separate a mixture, using the gas chromatograph, and then to determine from its chromatogram the identity of each alcohol and the mass percent of each alcohol in that mixture.

SAFETY

Goggles

Caution: Injection port is hot.

Syringes are very delicate and sharp. They must be handled with extreme care.

The various controls on the instrument have been preset for you.

**DO NOT MAKE ADJUSTMENTS!!!!!!!!!**

PRE‑LAB QUESTIONS

1a. Look up the chemical formulas, molecular weights, and boiling points for each of the following 3 compounds. These alcohols will be in the sample you will be investigating.

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
|  Alcohols | ChemicalFormula |  Mol. Wt.  (g/mol) | B.P. (°C ) | F.P. (°C)  |
| A. 1-butyl alcohol |  |  |  |  |
| B. 1-propyl alcohol |  |  |  |  |
| C. isopropyl alcohol |  |  |  |   |

 b. Predict the order of separation based on both molecular weights and boiling points.

 c. The mixture you will be analyzing will be carried through the column by the mobile phase. Identify the mobile phase.

2. Determine the retention times of each alcohol given the chromatogram of each known alcohol.

 a. Using a metric ruler, draw the baseline of the chromatogram.

 b. Measure the distance from the middle of the air peak to the middle of the alcohol peak. Record these measurements in the table below.

 c. Calculate the retention time of each peak and record in the table below.

|  |  |  |
| --- | --- | --- |
|  Alcohols | Distance (cm) | Retention Time (min) |
| A. 1-butyl alcohol |  |  |
| B. 1-propyl alcohol |  |  |
| C. isopropyl alcohol |  |  |

MATERIALS

 Gas Chromatograph

 10 uL syringe

 Samples A,B,C,D, which contain mixtures of varying percent of all or some of the following alcohols:

 1-butyl alcohol

 1-propyl alcohol

 isopropyl alcohol

PROCEDURE

1. You will work in teams of two or three.

2. Each team will quietly work on the PRE‑LAB.

3. Your teacher will notify your team when it is time to use the gas chromatograph.

4. You will be assigned an unknown mixture. Record the letter of this mixture in the appropriate

 space on the data table.

CLEANING THE SYRINGE

5. Clean syringe by inserting the needle into sample and draw up 5 - 10 ul. Depress the plunger of the syringe to discard the sample onto paper towel as described by your instructor.

6. Repeat step 5.

PREPARATION FOR INJECTING SAMPLE

7. Draw 10 ul of the sample into the syringe.

8. Onto the paper towel, depress the plunger to exactly 1ul. (No more than 1 ul should be

 injected into the G.C.)

9. Carefully pull back the plunger to add 5ul of air to the contents of the syringe.

INJECTION OF SAMPLE

10. Carefully insert needle into the injection port. **REMEMBER, THE INJECTION PORT IS**

 **VERY HOT**. Quickly depress the plunger and remove the syringe.

11. Look at the chromatogram as it is being printed. Your chromatogram should begin with an

 air peak and end with a flat, continuous baseline.

12. When chromatogram is completed, remove it from the chart recorder.

13. Return to your work area.

DATA ANALYSIS AND CALCULATIONS

14. Disregarding the air peak, label each successive peak at its top, beginning with #1, #2,...

 Record each peak number in the appropriate space on the data table.

15. Using a metric ruler, draw the baseline of the chromatogram.

16. Measure the distance from the middle of the air peak to the middle of the alcohol peak. Record these measurements in Data and Calculations Table #1.

17. Calculate the retention time of each peak and record in Data and Calculations Table #1. Show your work!!

18. Compare the retention times of your sample with the retention times of the known alcohols from the pre-lab to identify the alcohol that is producing each peak. Record the identity of the alcohol in Data and Calculations Table #1.

DATA AND CALCULATIONS TABLE #1 **Identity of unknown \_\_\_\_\_\_\_\_\_\_**

|  |  |  |  |
| --- | --- | --- | --- |
| Peak # | Distance (cm) | Retention Time (min) | Identity of Alcohol |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |

18. Measure the peak height and the peak width to the nearest hundredth of a centimeter

 for each peak. Record these measurements in the appropriate space in the Data and

 Calculations Table #2.

19. Calculate the area of each peak in cm2. Do this by multiplying ½ times the peak height

 times the width of the base (A = ½ Bh). Record your answers in the appropriate space on

 the Data and Calculations Table #2. Show your work!!

DATA AND CALCULATIONS TABLE #2

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Peak # | Identity of Alcohol | Peak Height at Max (cm) | Width of Base (cm) | Area of Peak(cm2) |
|  |  |  |  |  |
|  |  |  |  |  |
|  |  |  |  |  |

20. Calculate the total area in cm2 of all peaks by adding the area of each peak together. Record

 the total area in the space provided in the Data and Calculations Table #3.

21. Calculate the mass percent for each component of your mixture. For each

 component, divide the peak area by the total area and multiply by 100. Record these values

 in Data and Calculations Table #3.

DATA AND CALCULATIONS TABLE #3

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Peak # | Identity ofAlcohol | Area of Peak(cm2) | Total Area of Peaks(same for all) | PercentComposition |
|  |  |  |  |  |
|  |  |  |  |  |
|  |  |  |  |  |

CONCLUSIONS

1. Which physical property, B.P. or molecular weight, accounted for the distribution of peaks on

 the chromatograph? Explain.

2. On the baseline of each peak of the chromatogram, write the name of the most likely

 alcohol indicated by that peak, and the percent composition calculated for that compound.

3. How could you verify your answers to conclusion 2 (above) using the gas chromatograph?

4. A mixture of ethyl alcohol (molecular weight 46.07 g/mol, B.P. 75°C), 4-chloro-butanone

 (molecular weight 90.55 g/mol, B.P. 75°C) and diisopropyl N-nitrosoamine (molecular weight

 130.19 g/mol, B.P. 76-81°C) was injected into a G.C. Predict the order in which the

 substances would appear on the chromatogram.

5. Examine your chromatogram.

 Three inferences which can be made from your chromatogram are:

 1.

 2.

 3.



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