

GAS CHROMATOGRAPHY

PURDUE UNIVERSITY INSTRUMENT VAN PROJECT

DETERMINING THE PERCENT COMPOSITION OF A MIXTURE OF VOLATILE LIQUIDS USING A GAS CHROMATOGRAPH.

(Revised: 1-12-93)

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 percent composition of each component in the mixture can be determined. 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 one of the following methods:

- * The peak can be treated as a triangle and the formula, $\text{Area} = \text{height} \times \text{width at } 1/2 \text{ height} \times \text{attenuation}$ can be applied.
- * Each peak can be cut out and weighed.

There is one factor in determining the areas which must be considered when using a thermal conductivity detector. The thermal conductivity of different substances tends to vary slightly. This means that the response of the thermal conductivity detector will also vary slightly. Therefore a correction factor will be used for each component of the mixture to account for this conductivity difference.

After finding the corrected areas, the percent composition for each peak is calculated by:

1. Adding together the peak areas,
2. Dividing each area by the total area and multiplying by 100.

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PURPOSE

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

SAFETY

Goggles/aprons

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 4 compounds. Some or all of these may be in the sample you will be investigating.

Compounds	Chem. Formula	Mol. Wt. (g/mol)	B.P. (°C)	F.P. (°C)
A. 1-butyl alcohol				
B. t-butyl alcohol				
C. 1-propyl alcohol				
D. 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.

- 2a. Read the Scientific American article, "Gas Chromatography," by Roy A. Keller. (Keller, Roy A. (1961). Gas Chromatography. Scientific American, 4, 58-67.) Summarize the history of the gas chromatograph in the minimum of four well-developed paragraphs.

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MATERIALS

Gas Chromatograph
10 μ L Hamilton syringe
Samples A,B,C,D, which contain mixtures of varying percents of all or some of the following volatile compounds:
1-butyl alcohol
t-butyl alcohol
1-propyl alcohol
isopropyl alcohol

PROCEDURE

1. You will work in teams of two.
2. Each team will quietly work on parts 1 and 2 of 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 μ L. 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 μ L of the sample into the syringe.
8. Into the waste container, depress the plunger to exactly 1 μ L. (No more than 1 μ L should be injected into the G.C.)
9. Carefully pull back the plunger to add 5 μ L of air to the contents of the syringe.

INJECTION OF SAMPLE

10. Prior to injection, check the baseline on the recorder for proper adjustment. If it is not properly aligned, obtain help from your instructor.
11. Carefully insert needle into the injection port. **REMEMBER, THE INJECTION PORT IS**

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

12. Look at the chromatogram as it is being printed. Your chromatogram should begin with an air peak and end with a flat, continuous baseline. On your chromatogram take down the following information:

Attenuator setting
Column temperature
Sample size
Identity of sample

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

14. Return to your work area.

15. 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.

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

17. Measure the peak height and the peak width at 1/2 height to the nearest tenth of a centimeter for each peak. Record these measurements in the appropriate space on the Data/Calculation Table #1.

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DATA / CALCULATIONS TABLE #1

Identity of Unknown _____

Peak #	Peak Identification	Peak Height (cm)	Width at 1/2 Peak Height (cm)	Attenuation	Area (cm ²)

CALCULATION TABLE #2

Peak #	Peak Identification (from above)	Area (cm ²) (from above)	Correction Factor	Corrected Area (cm ²)

CALCULATIONS SHOW ALL WORK!

1. Calculate the area of each peak in cm². Do this by multiplying the peak height times the width at 1/2 peak height times the attenuation. Record your answers in the appropriate space on the Data/Calculation Table #1.
2. Correct each peak area using the correction factor for each substance given in Reference Table #1. Multiply each peak area by the correction factor for that substance. This is the corrected peak area, and it should be recorded on Calculation Table #2.

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REFERENCE TABLE #1

Substance	Correction Factor
1-butyl alcohol	1.000
t-butyl alcohol	1.000
1-propyl alcohol	0.920
isopropyl alcohol	0.903

3. Calculate the total area in cm^2 of all peaks by adding the corrected areas. Record the total area in the space provided in the Calculation Table #3.

CALCULATION TABLE #3

Peak #	Peak Identification	Total Peak Area (same value for all)	Percent Composition

4. Calculate the mass percent for each component of your mixture. For each component, divide the corrected peak area by the total area and multiply by 100. Record these values in Calculations Table #3.

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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 compound 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?

FOLLOW-UP QUESTIONS

1. 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.

2. Examine your chromatogram.

Three inferences which can be made from the chromatogram are:

- 1.
- 2.
- 3.

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TEACHERS' GUIDE DETERMINING THE PERCENT COMPOSITION OF A MIXTURE OF VOLATILE LIQUIDS USING A GAS CHROMATOGRAPH

CLASSROOM USAGE Chemistry I

CURRICULUM INTEGRATION

Solutions
Percent composition
Physical properties
Molecular weight
Bonding/ Polarities

PREVIOUS KNOWLEDGE

Metric measurement
% calculation
Significant digits
Definition of solution
Molecular weight
Boiling point
Operations of a gas chromatograph and underlying principles.

PREPARATION The teacher should use a sample chromatogram to instruct students on mathematical calculations for area determinations of peaks and % composition of a mixture. It is suggested that students be given a practice problem for guided instruction the day prior to the lab. Use a chromatogram which you have obtained previously.

A major consideration in determining retention time in chromatography is polarities. In this experiment, the polarities of all the substances are very similar. Due to this, the students are led to looking at molecular weights and boiling points as other considerations.

In this case of gas chromatography, the column (stationary phase) is carbowax. Carbowax is a slightly polar solid. The mobile phase is helium.

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TIME Each sample takes approximately 10 minutes to run through the G.C.

SUGGESTION Have students work in rotations. Some groups will be using the CRC, others will be using the G.C., while others work on the history of the G.C.

SAFETY AND DISPOSAL

Students should be warned of the hazards of the syringe needle and the hot injection port. Solutions should be disposed of according to Flinn disposal 27J – evaporation in a fume hood.

PRE-LAB ANSWERS

1a. Data Table:

	Chem. Formula	Mol. Wt. (g/mol)	B.P. (°C)	F.P. (°C)
1-butyl alcohol	CH ₃ (CH ₂) ₃ OH	74.12	117.7	-90
t-butyl alcohol	(CH ₃) ₃ COH	74.12	83	25
1-propyl alcohol	CH ₃ (CH ₂) ₂ OH	60.10	97	-127
isopropyl alcohol	CH ₃ (CH ₂) ₂ OH	60.10	82.4	-89.5

b. isopropyl alcohol, t-butyl alcohol, 1-propyl alcohol, and 1-butyl alcohol

c. helium

2a. If computers are available, students can word-process their reports.

ANALYSIS

There are two ways to determine the identity of each peak. One way is to run a chromatogram of each alcohol standard and use retention time. The other way is to spike the mixture with each standard and observe which peak increases in area.

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CONCLUSIONS

1. Boiling point. Because molecular weights are all approximately the same, the separations must have been due to the differences in boiling points.
2. Refer to student's chromatogram.
3. By running a pure sample of each component.

FOLLOW-UP QUESTIONS

1. Ethyl alcohol, 4-chloro-butanone, diisopropyl n- nitrosoamine.
2. 1. 3 substances in the mixture
2. each component may have different boiling points
3. each component may have different molecular masses.
4. other possible answers may apply.

ASSESSMENT

A. Checklist:

1. Use print-out, chromatograph to access the proper injection of a sample

B. Activities:

1. Have a chromatograph set up containing an air peak followed by 2 peaks. Have areas of each peak already calculated - they are to calculate the mass percent composition for 1 of the peaks.

C. Final-portfolio –

1. Report on the history of the GC