# Distillation and Gas Chromatography

Distillation is a technique often used to purify a liquid or to separate liquid components of a mixture. Essentially, it is the process of converting a liquid (called the distilland) to vapor by heating it to the boiling point and condensing the vapor back to a liquid (called the distillate). If applied to a mixture of liquids, the vapor (and thus the distillate) will be enriched in the more volatile component in the early part of the distillation. If the components of the mixture have sufficiently different boiling points, they can be separated by distillation.

Gas chromatography (GC) is a method of analysis that separates the components of a mixture based on their relative boiling points. A sample is vaporized and carried through a column containing a liquid stationary phase by a gaseous mobile phase (usually He). For most GC analyses, partitioning between the stationary liquid phase and the gaseous mobile phase is based on the boiling point of the material (more volatile materials move faster through the column), and therefore the separation of a mixture by GC is usually based on the relative boiling points of the components. Lower boiling compounds generally travel through the column faster than higher boiling compounds. Thus, lower boiling compounds generally have lower retention times than higher boiling compounds. The composition of the mixture can be determined from relative peak areas, but since the detector response is different for different compounds, the peak areas from the chromatogram must be corrected by a response factor before quantifying the composition of any sample. To correct your peak areas, divide the peak area from the chromatogram by the appropriate response factor. Typical component response factors (on the GowMac GCs used for this lab) are: hexane (1.50), cyclohexane (1.80), heptane (1.63), toluene (1.41), ethylbenzene (1.00).

In this experiment, you will be assigned an unknown mixture consisting of two of the liquids from the table below. You will separate the mixture by both simple and fractional distillation, and in the process, determine the boiling point of each component in your unknown mixture. In addition, you will analyze distillate samples at the beginning and the end of the distillation by gas chromatography. Using this data, you will determine the identity of the components of your unknown mixture, and comment on the effectiveness of each distillation technique.

**Required Reading (Padias):**

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| **Topic** | **2nd Edition** | **3rd Edition** |
| Distillation | pp. 141 – 155 | pp. 143 – 157 |
| Chromatography, general | pp. 162 – 163 | pp. 164 – 165 |
| Gas Chromatography | pp. 179 – 187 | pp. 181 – 189 |

**Safety:**

The unknown samples are mixtures of flammable solvents. Keep away from spark sources and open flames.

Do not distill to dryness! A dry flask may crack if heated to hot.

The steel wool used as the column packing can cut into skin. Handle with care.

Use a boiling chip when heating liquids to prevent “bumping”

***Injector ports on a gas chromatograph are hot!*** Avoid contact with the GC injector ports.

*NOTE: This experiment is to be done in groups of 4 students. Each group will be assigned an unknown. Half of the group will perform the simple distillation, and the other half will perform the fractional distillation. Students must share their procedure, observations, and data with all members of the group. Each student is then required to write an individual lab report.*

**Procedure:**

Simple and Fractional Distillation of the Unknown Mixture

Assemble the distillation apparatus (simple or fractional) using a 10-mL round bottom flask, distillation column (fractional only), distillation head, condenser, take-off adapter, 3-mL conical vial and a thermometer with adapter as shown below. Be sure that the thermometer bulb is just below the side arm of the distillation head. Place a boiling chip in the round bottom flask and add 7.0 mL of the unknown mixture. Turn on the cooling water to the condenser and heat the mixture to a boil and using the hotplate with an aluminum heating block. As the hot vapors begin to heat the glassware, you should see a “reflux ring” of condensate begin to rise into the distillation head. When the reflux ring reaches the thermometer bulb, you will see a rapid rise in temperature up to the boiling point of the distillate. The vapor will condense in the condenser and begin to drip into the conical vial. Adjust the heat so that about 1 drop of distillate is collected every 5 seconds. Beginning with the first 0.5 mL of distillate, record the vapor temperature at every 0.5-mL interval throughout the distillation.

  

 **Simple Distillation** **Fractional Distillation**

When you have collected 1.0 mL of distillate, remove and cap the conical vial, and label with your notebook number and “Initial Sample.” Collect an additional 3.5 mL of distillate in a 2nd, 5-mL conical vial. After you have collected **a total of 4.5 mL** of distillate, collect the remainder of the distillate (until about 0.5 mL is left in the distillation flask) into a 3rd clean, dry conical vial. When you are finished, cap the vial containing this final distillate and label with your notebook number and “Final Sample.”

Gas Chromatography

Inject 1 L of sample into the GC (your instructor will demonstrate). Depending on the effectiveness of the distillation, you may see either one or two peaks. Your instructor will provide you with a reference chromatogram to help you identify the components of your unknown by their retention time. Integrate the chromatogram to get the peak areas (your instructor will demonstrate the use of the software).

**Possible Unknown Components**

|  |  |
| --- | --- |
| *Compound* | *bp (°C)* |
| Hexane | 69.0 |
| Cyclohexane | 80.7 |
| Heptane | 98.4 |
| Toluene | 110.6 |
| Ethylbenzene | 136.0 |

**Reference:**

Pavia, D. L.; Lampman, G. M.; Kriz, G. S.; Engel, R. G. *Introduction to Organic Laboratory Techniques, A Microscale Approach;* 3rd ed.; Brooks/Cole: Pacific Grove, CA, 1999; pp. 51 – 56.

1. Unknown number: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

*Distillation Data*

2. Construct a graph of vapor temperature (y-axis) vs. volume of distillate (x-axis) for both the simple and the fractional distillations. Attach the graphs to this report.

3. Determine the boiling points for the components of your mixture using the *fractional distillation* graph. Indicate on the graph how you made your boiling point determination.

*Component 1 bp:* \_\_\_\_\_\_\_\_\_\_\_\_\_ *Component 2 bp:* \_\_\_\_\_\_\_\_\_\_\_\_\_

4. Identify the components of your mixture based on their boiling points.

*Component 1:* \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ *Component 2:* \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

*Gas Chromatography Data*

5. Attach the GC chromatograms for each distillation sample to this report.

6. Record the information below for each component in your samples. Identify each component using its retention time and calculate the % of each component in your samples(*show your calculations in the space on the next page*):

|  |  |  |  |
| --- | --- | --- | --- |
| Sample | Retention Time | Component Identity(based on retention time)  | % in Sample |
| Simple - Initial |  |  |  |
|  |  |  |
| Simple - Final |  |  |  |
|  |  |  |
| Fractional - Initial |  |  |  |
|  |  |  |
| Fractional - Final |  |  |  |
|  |  |  |

*Worksheet Item #6 Calculations:*

***NOTE****: Since the detector response is different for different compounds, the peak areas from the chromatogram must be corrected by a response factor before quantifying the composition of your sample. To correct your peak areas, divide the peak area from the chromatogram by the appropriate response factor. Typical response factors for the possible components are as follows: hexane (1.50), cyclohexane (1.80), heptane (1.63), toluene (1.41), ethylbenzene (1.00).*

*Conclusions*

7. Provide a final identification of the components in your mixture, and fully explain how you came to this conclusion. If the boiling point data and GC retention time data gave conflicting results, justify your final identification.

*Component 1:* \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ *Component 2:* \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

8. Discuss which distillation method provided better separation of your components.

**Questions**

**(Fully explain how you arrived at each answer, including all calculations.)**

1. Consider the following pairs of compounds. For which pairs can simple distillation be used, and for which ones would fractional distillation be more suitable? Explain your reasoning.

a) hexyl acetate and butyl acetate

b) 1-hexanol and cyclopentanol

2. A mixture of 1-bromobutane, 2-bromobutane and 2-chlorobutane was injected into a gas chromatograph and gave the chromatogram below. Identify the peaks and explain how you made your identification. Determine the approximate composition of the sample (assume the three compounds have equal response factors). Show all calculations.



3. Vacuum distillation a technique often used to purify compounds with high boiling points, because the boiling point of a compound is lower when a vacuum is applied than it is at atmospheric pressure. (a) Why are boiling points lower under vacuum than at atmospheric pressure? (b) If a compound boils at 300 °C at atmospheric pressure, what is its boiling point at 1.0 torr? Explain how you determined your answer.