Sample Lab Report

Simple and Fractional Distillation

Unknown # 2

Purpose

In this experiment we aim to demonstrate that we can separate two volatile compounds from a mixture due to the different chemical properties of each compound. We will accomplish this by a separation procedure known as distillation, which relies on each compound having a distinct and separate boiling point. Our pure products will be analyzed with gas chromatography to determine the success of the distillation.

Procedures

The experiment was performed as stated in the course textbook:

> Pavia, D. L., Lampman, G. M., Kriz, G. S., Engel, R. G. *Introduction to Organic Laboratory Techniques: A Microscale Approach*. **2007**, 4th Ed. Pp **51—57**. .

Data

The distillation curves for our simple and fractional distillation (See page 3) clearly demonstrate that fractional distillation separates the two compounds more completely. The boiling point (bp) of our unknown compounds was taken from the flat regions of the fractional distillation curve. Our unknown mixture contained hexane (bp 69 ºC) and toluene (bp 110.6 ºC).

Analysis via gas chromatography allowed us to determine the relative percentage of hexane and toluene at fractions near the beginning and end of our distillations. Relative percentages have been recorded in the table below, and our calculations are shown on page 5.

	Simple Distillation Retention time (s)		Fractional Distillation Retention time (s)	
	Cyclohexane Retention time (s)	Toluene Retention time (s)	Cyclohexane Retention time (s)	Toluene Retention time (s)
Fraction 1 (4th mL)	15	25	14	24
Fraction 2 (17th mL)	14	26	16	25

	Simple Distillation Area (s)		Fractional Distillation Area	
	Cyclohexane Retention time (s)	Toluene Retention time (s)	Cyclohexane Retention time (s)	Toluene Retention time (s)
Fraction 1 (4th mL)	15551	400	14444	248
Fraction 2 (17th mL)	222	16558	166	25995

A figure of the gas chromatography data has been included on page 4.

Results and Calculations

Simple Distillation Fraction#1

Cyclohexane Total Area $15551 + 400 = 15951$ % Cyclohexane = 15551/15951 X100 = 97.49232%

%Toluene = 400/15951 X 100 = 2.5077 %

Simple Distillation Fraction#2

Cyclohexane Total Area $222 + 16558 = 16780$ % Cyclohexane = 222/16780 X100 =1.3230%

%Toluene = 15951/16558 X 100 = 96.334 %

Fractional Distillation

This calculation was done the same as the Simple Distillation

Fraction #1 (4mL)

 %Cyclohexane 98.312%   %Toluene 1.5548

Fraction #2 (17mL)

 %Cyclohexane 0.63453%   %Toluene 99.365%

Fractional Distillation

Calculation similar to Simple Distillation %

 % Cyclohexane

 % Toluene   = 0.63453%

Conclusion

This week we utilized two methods of distillation (simple and fractional) to separate a mixture of two volatile compounds. We found that while the simple distillation separated the majority of the two compounds near the beginning and the end of the distilling process, fractional distillation produced much more pure fractions. In simple distillation the column was shorter, allowing less room for the two different compounds to fully separate. While heating the round-bottom flask the hexane molecules gain higher kinetic energy faster than the toluene molecules due to their lower molecular weight and lesser intermolecular forces. The longer fractional distillation column allowed the hexane molecules with higher kinetic energy to separate from the lower-energy toluene molecules. After viewing the gas chromatograph data from the fractional distillation we saw that our unknown compounds had almost completely separated, while the fractions from the simple distillation were less pure.

Although distillation is a good separation technique it is still necessary to ensure that proper steps are taken to reduce the chances of error. Failure to add a boiling chip to the round-bottom flask could allow the mixture to heat unevenly. This could allow molecules with a higher boiling point to gain kinetic energy before the molecules with the lower boiling point, which would create impure fractions. Watching the rate of temperature increase is also important. Allowing the temperature to increase too quickly can cause impurity for the same reason. We forgot to add the boiling chip to the round-bottom flask in the simple distillation, which could have contributed to the mixture of toluene and hexane that we saw. We made sure to include the boiling chip in the fractional distillation, however, and did not observe any problems.

Even though both hexane and toluene are volatile, they have different chemical properties to allow for separation and analysis. Hexane is a hydrocarbon with no dipole moment or double bonds. This means that it has few intermolecular interactions, and will have a lower boiling point. It also has a lower molecular weight than toluene, so it will appear first on the gas chromatograph. Toluene has a benzene ring with a methyl group attached. Toluene has greater intermolecular forces than hexane because the benzene ring withdraws electrons from the methyl group. This creates a slight polarity to the molecule that increases intermolecular forces and increases the boiling point. Since toluene has a greater molecular weight than hexane it will appear second on the gas chromatograph. This experiment illustrates how useful the different properties of compounds can be while trying to purify mixtures of compounds.
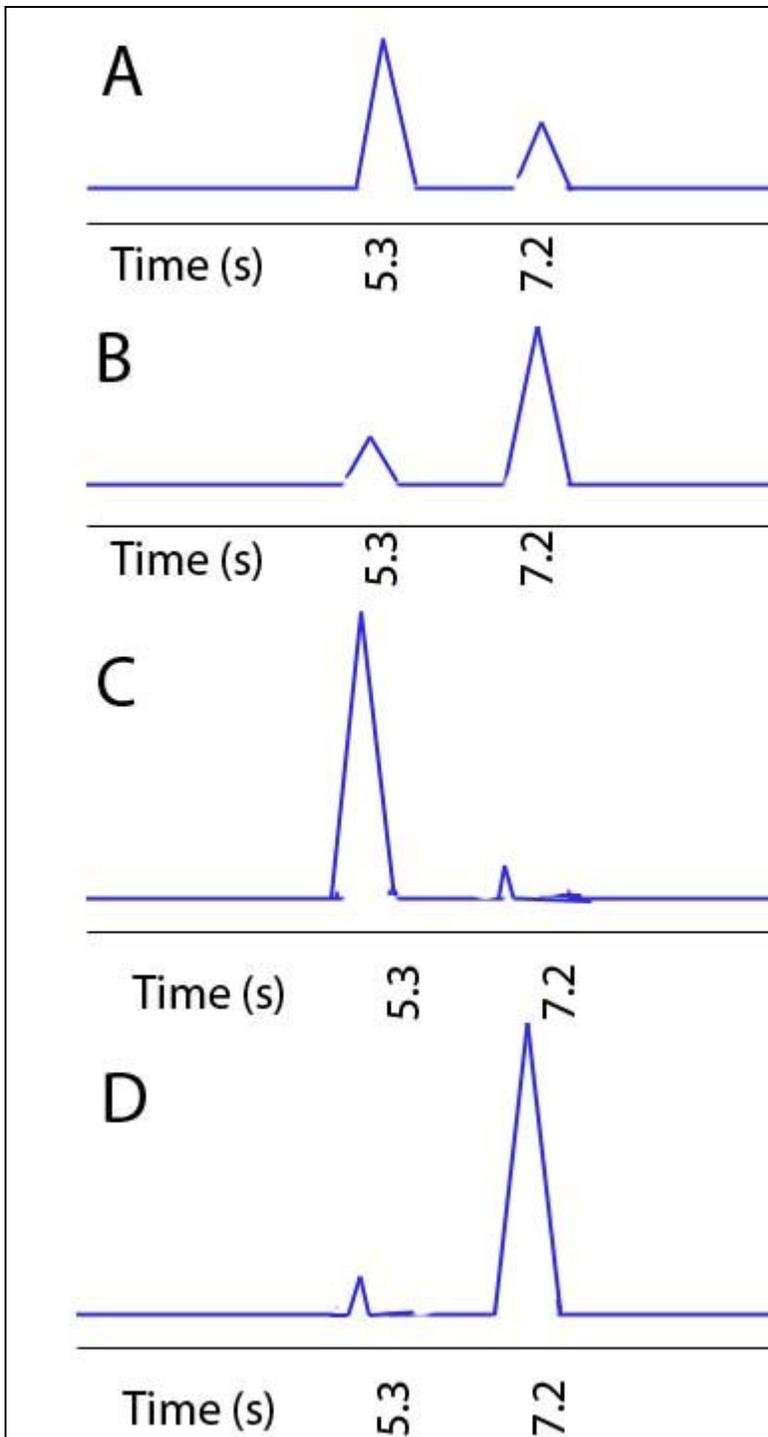
NOTE: Graphs should take up at least a half page. The Data shown below has been fabricated for the purposes of demonstration and should not reflect what you actually would get in the lab.

Separation of Unknown with Simple Distillation
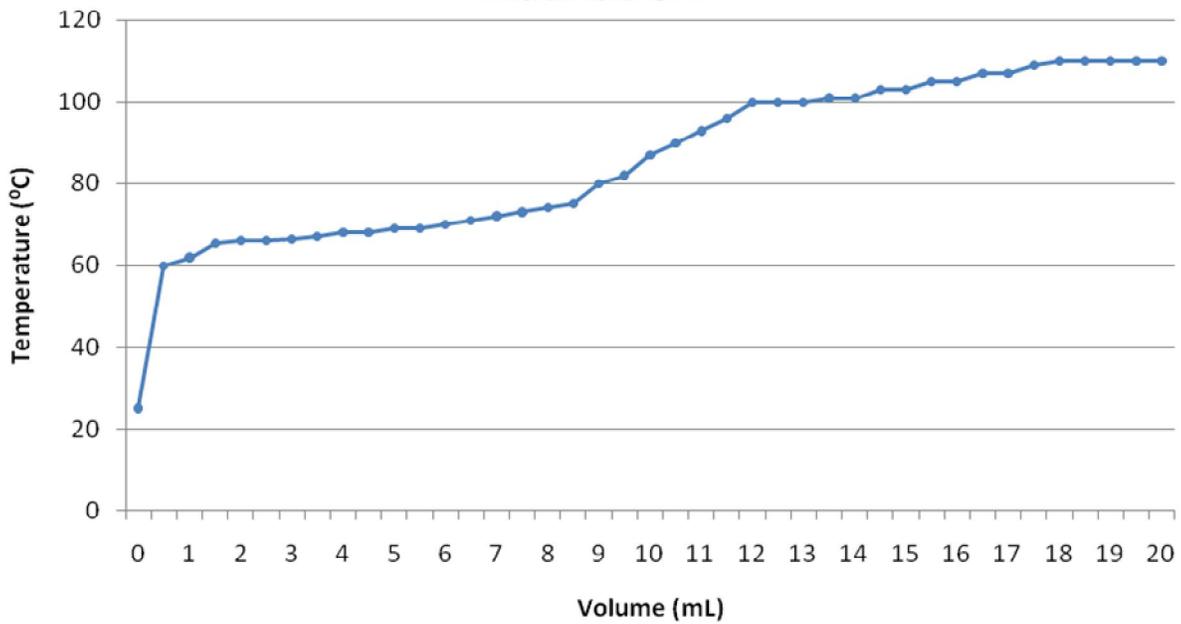
Separation of Unknown with Fractional Distillation
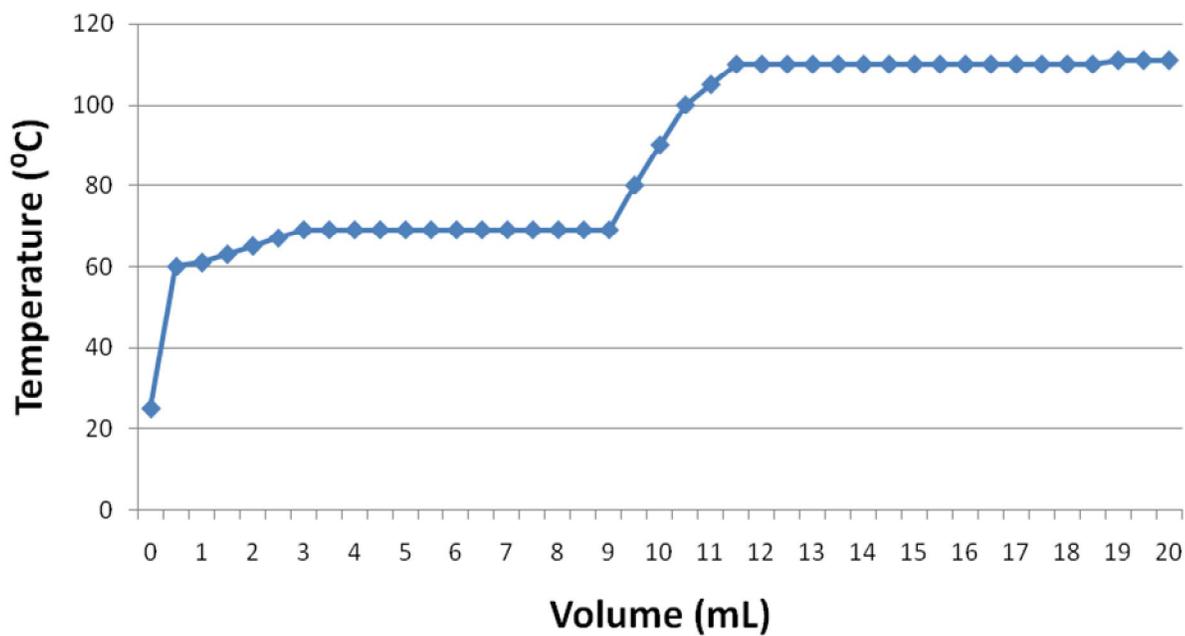
Gas Chromatography Data from the Simple and Fractional Distillations. Hexane passes through the column at 5.3 seconds and toluene passes through the column at 7.2 seconds.

A. 4th mL fraction from simple distillation. Hexane is more abundant than toluene.

B. 17th mL fraction from simple distillation. Toluene is more abundant than hexane.

C. 4th mL fraction from fractional distillation. Fraction is nearly all hexane.

D. 17th mL fraction from fractional distillation. Fraction is nearly all toluene.

<u>Sample Calculations (For 4th fraction of simple distillation)</u>

Amount of compound is directly proportional to the area under the gas chromatography peak:

Area $= \frac{1}{2}b * h$, where b is the base of the peak and h is the height of the peak.

Area Hexane $= \frac{1}{2}(2mm * 3mm) = 3\text{mm}^2$

Area Toluene $= \frac{1}{2}(1mm * 2mm) = 1\text{mm}^2$

Percent Hexane $= \frac{area\ Hexane}{sum\ of\ areas} = \frac{3mm2}{4mm2} * 100\% = 75\%$ Hexane

Percent Toluene $= \frac{area\ Toluene}{sum\ of\ areas} = \frac{1mm2}{4mm2} * 100\% = 25\%$ Toluene