

Simple and Fractional Distillation

CHEM 315
Week of 10/25/10
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Due dates

- Today
 1. Gas Chromatography lab report - at the beginning of class
 2. Spectroscopy problem set, Part 2, problems 3, 4 and 5
 3. Carbon copies for Simple/Fractional Distillation experiment - at the end of class
- Next week
 1. Simple/Fractional Distillation lab report - at the beginning of class
 - See <http://classweb.gmu.edu/chemlab/sws/315/distill-report.htm> for instructions on what to include in your report

Purpose

- Today you will compare the separating efficiencies of:
 1. simple distillation with no column, and
 2. fractional distillation using an unpacked Vigreux column
- The mole percent composition of the mixture (ethyl and butyl acetate) is unknown
 - You will use what you learned from the GC lab last week to determine the mole percent composition

Simple vs. Fractional

- You would use simple distillation if
 1. the boiling points of the components differ by greater than 100 deg C, or
 2. the impurity is less than 10%
- You would use fractional distillation if
 1. the boiling points of the components in the mixture are similar (less than 100 deg C)
 2. the distillation is longer with more vaporization-condensation cycles
 - this is like re-distilling over and over again
 - each cycle will enrich the composition of the vapor with the compound with the lower boiling point

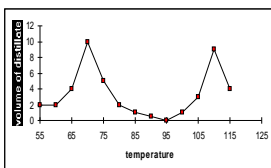
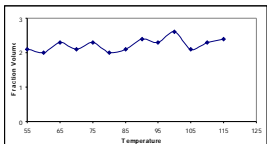
Experiment - procedure

1. Set up the distillation - decide which group member will do the fractional and simple distillation
 - Remember that I must approve of your set-up before you begin!
2. Put mixture of unknown EtOAc and BuOAc composition in your flask with the boiling stones
3. Carry out your distillation slowly but steadily
 - Slow distillation ensures that many vaporization-condensation cycles occurs, thereby maximizing the purity of each fraction
4. Collect the fractions in test tubes
 - Collect liquid that distills over a 5 deg C interval into the same test tube; don't worry too much about the resultant volume
5. Measure the volume of each fraction

Experimental - data analysis

- You will be making a graph of the volume of fraction vs. temperature
 - Expect to collect the most volume at the temperature(s) closest to the boiling point
- I recommend making a line graph using Excel for this

Due dates



- You will analyze these graphs in a similar fashion to your GC chromatographs (remember method of triangulation?) to determine the mole percent of your unknown mixture

Final notes

- Measure the volume of your unknown mixture using a graduated cylinder
 - The fraction volumes should add up to a value close to your starting volume
- To maintain temperature, wrap your column with aluminum foil (fractional distillation only)
- Remember to let me approve of your set-up before you start
- You may need higher heat settings to reach the b.p. of BuOAc
- Aim for a distillation of ~ 1 drop/sec
- Collect the fractions in test tube over ICE!
- Do not discard the fractions; you're going to need them!

Final notes, con't

- Working in groups
 - Groups of 2 students
 - One student does fractional distillation and the other student does simple distillation
 - No member of the group may leave the lab until the distillations are completed and the data has been recorded in everyone's lab notebook
 - Be sure to include the name of your group member on your notebook copy too
 - Make sure that everyone in the group knows and understands exactly what happened during each of the experiments (fractional and simple distillation) and the resultant data.
 - Exchange data with your group members and jot this down in your notebook before you leave
 - It will be your fault if you don't understand your data, whether or not you did the experiment**
- After writing your lab report, make a copy of it. You will need it for reference for next week's experiment: Gas Chromatography of Distillate Fractions
