

Chemistry 143: Fractional Distillation and GC Analysis

1. Fractional Distillation

- *Read pages 62-78*

Possible Unknowns

cyclohexane

water

chloroform

diethyl ether

hexane

2-propanol

ethyl acetate

toluene

methanol

ethanol

*These chemicals should be listed in your table of physical constants with their boiling points, densities, and molecular weights. You should **draw the structures** in the equation portion or your pre-lab.*

Obtain an unknown sample. Record the unknown number/letter.

Assemble a fractional distillation apparatus. This is the same as the simple distillation apparatus, except that you should insert a fractionating column (see p.72) between the boiling flask and the Claisen adapter. Use a 250 mL round bottom flask (RBF) as the boiling flask and a graduated cylinder in place of the collection flask. Clamp the whole apparatus securely to ring stands.

Add your unknown mixture (save ~1 mL for GC analysis) to the 250 mL RBF and add a couple of boiling stones. Turn the variac up about half way. Turn on the condenser cooling water and wait for the liquid in the flask to begin boiling, at which time the heat should be turned down substantially such that the vapors travel very slowly up the fractionating column. Follow this visually by looking at the condensing ring slowly moving up the fractionating column.

Construct a data table of temperature and volume of distillate. Make measurements every 1-2 mins. The temperature should plateau for a while as the first component distills. The distillation should slow or stop (the temperature may drop briefly), then the temperature will begin to rise again. When the temperature reaches a second higher plateau, begin to collect the second

component in a separate graduated cylinder. Continue to make measurements until ~95% of the second component has been distilled. Record the total volume of both components. Turn off the variac and lower the heating mantle.

Using a spreadsheet program plot a graph of temperature vs. volume of distillate, and tape the graph into your notebook. Determine the boiling points of your two components. Identify the two unknowns.

2. Gas Chromatography

Obtain 3 gas chromatograms for your report. They should be the original unknown sample and the two separated components. Tape the chromatograms into lab notebook. Comment on the purity of your distilled components.