12AL Experiment 5: Fractional Distillation

Instructor Note: Day 1 (half of the class); Day 2 (other half); limited macro distillation equipment & organic students must perform on an individual basis.

Safety: Proper lab goggles/glasses must be worn (even over prescription glasses). Distillation of organic mixtures releases irritating and sometimes toxic vapors, and often times, volatile solvents splatter vigorously out of their containers! As always, ask where organic waste containers are located in the lab.

Background:

Distillation is a physical method of separating mixtures based on differences in volatility while undergoing vaporization/boiling. Thus, it is important to continue to understand how the intermolecular attractions of a substance affect its volatility.

Distillation has widespread uses such as the separation of crude oil into more pure fractions for specific uses such as gasoline, diesel, and kerosene. Seawater can be distilled to remove salt; air can be separated into pure oxygen and nitrogen gases etc.; alcohol mixtures are distilled to increase the alcohol percentage; and in organic chemistry, distillation is used to isolate & extract components from a mixture, as well as to purify compounds isolated or synthesized. (Wikipedia)

In distillation, a mixture is slowly heated in a round-bottom flask containing a boiling chip. The component of the mixture with the weakest intermolecular forces and therefore, the lower boiling point, will vaporize first – its vapor travels up a column until it reaches the cool air of the condenser, where the vapor will condense back into its liquid state and drip out the end of the condenser into a collection container (see diagram). This process continues with each compound in the mixture, from lower boiling point to higher boiling point; all that is required is for the chemist to switch out collection containers when there are "lulls" (slowing/pausing) in the dripping out of liquid. Distillations are slow and take great patience. It is important for the chemist to pay constant attention to prevent the recollection of a mixture – the goal is to collect pure substances.

Organic chemists perform two types of distillation, simple and fractional distillation. Simple distillation utilizes a simple vaporization column, while fractional distillation utilizes a fractionated containing barriers up the column called theoretical plates ("glass teeth," aluminum, beads) on which the refluxing liquid can condense, re-evaporate, and condense again, essentially distilling the compound over and over. Simple columns are used to separate mixtures whose components are farther apart in boiling point, while fractionating columns are used when compounds have close boiling points generally being separated by 10˚C or less.

Objective: To learn how to set-up a fractional distillation apparatus, as well as to separate an organic mixture (alcohol/ketone) into its individual pure substances. To learn how to operate the IR spectrophotometer in order to check purity of your fractions collected (however, IR analysis will fully be investigated in the next experiment).

Procedure:

1. SLOWLY & CAREFULLY set up the fractional distillation apparatus. *Please note: deductions in lab grades will occur due to the breaking of distillation glassware – macro-distillation glassware is VERY EXPENSIVE and we have extremely LIMITED QUANTITIES. Please do not rush, do not chit chat with your friends during set-up, and never let go of a piece of glassware until you are absolutely, 100% sure, that it is CLAMPED securely to a metal bar. And ALWAYS, LUBRICATE all glass pieces that connect to one another with GLYCEROL every time (1 drop goes a long way!).

2. Measure out 20mL of the alcohol/ketone mixture. Pour into your round bottom flask and add a boiling chip.

3. Use a hot plate to heat the mixture. The round bottom flask should be touching the hot plate. Begin a low setting and slowly increase the heat until you are at a gentle boil – DO NOT start on a high setting and do not over boil – the goal is to separate the two compounds and vigorous boiling will cause both to shoot through the column and condenser to your collection container. Remember, distillation is a very slow process. In fact, most organic experiments are long and slow and it is very important to practice PATIENCE.

4. There are many different ways to collect fractions that distill over. If there are multiple substances to be collected, then the chemist should switch out collection containers when there are natural pauses in the rate at which the liquid is dripping out of the condenser – for example, the low boiling point substance will drip out first and then its rate will slow and often stop as the next higher boiling point substance travels out next, etc…

Since we are only concerned with separating two substances, the alcohol and the ketone, we will do the following: Collect about 1-2mL of the liquid that begins to drip out of the condenser; then switch out the collection beaker with a second beaker collecting the liquid that drips out until there just a few milliliters left in your round bottom flask. NEVER DISTILL TO DRYNESS – there should always be liquid remaining in flask.

What does our technique do? Well, the first 1-2mL should be PURE LOW BP SUBSTANCE, the second beaker should be a mixture, and the few mL left in your round-bottom flask should be PURE HIGHER BP SUBSTANCE (if not pure, you can further distill off some liquid and retest).

5. Run your two "pure" samples separately on the IR (do not run second beaker mixture)– your instructor should teach every individual student how to properly use the IR. Please pay attention and make sure to clean the IR trough with isopropyl alcohol BEFORE & AFTER you run your sample. Print each spectrum.

Pure Alcohol: should have characteristic 3400cm^{-1} stretch of the O-H bond
Pure Ketone: should have characteristic 1700cm^{-1} stretch of the C=O bond

*If your samples are impure, then you will see both the 3400cm^{-1} and 1700cm^{-1} stretches on the same IR – if this occurs, you need to REPEAT YOUR DISTILLATION.

6. Label your IR spectrums. Each spectrum should be labeled with Alcohol or Ketone and the O-H and C=O bonds should be labeled at their appropriate wavenumber on the IR. Attach to postlab.

12AL Prelab 5: Fractional Distillation

1. Distillation separates compounds in a mixture based on what criteria?

2. When is fractional distillation used?

3. What do you think would be the preferred distillation method, simple or fractional, if you could only ever use one method? Explain.

4. What is the exact definition of a boiling point?

5. What must happen exactly to the liquid state in order for the molecules to vaporize? Be clear and your answer must use the term "intermolecular forces."

12AL Postlab 5: Fractional Distillation

1. What type of molecules distill first – more or less volatile? Explain in terms of specific intermolecular forces & give an example not included in this postlab.

2. Distillations often have a thermometer that is placed at the top of the vapor column with the thermometer bulb just at the mouth of the condenser – do you think you should rely on the thermometer temperatures to separate your mixture? Why or why not? Explain clearly.

3. Look up the boiling points of the following substances, draw their structure, and label their functional groups (ie: ester, ketone, etc…):

Acetone

Butanamine

Butyl Acetate

Butanol

Cyclohexane

Cyclohexanol

Benzene

4. Should the following be separated by simple distillation? Briefly explain.

Butanol & Butanamine?

Butyl Acetate & Butanol?

Cyclohexane & Cyclohexanol?

Cyclohexane & Benzene?

5. In a distillation, which compound would distill last & why? Your answer must also indicate the intermolecular forces holding the substance in its liquid state for each substance AND their relative strength. If two substances have the same intermolecular forces, be clear in WHY one is stronger than the other.

Butanol & Butanamine

Cyclohexane & Benzene

6. Are your completely analyzed IR spectrums attached?