Reminder: These notes are meant to supplement, not replace, the textbook and laboratory manual.

Boiling Point and Index of Refraction

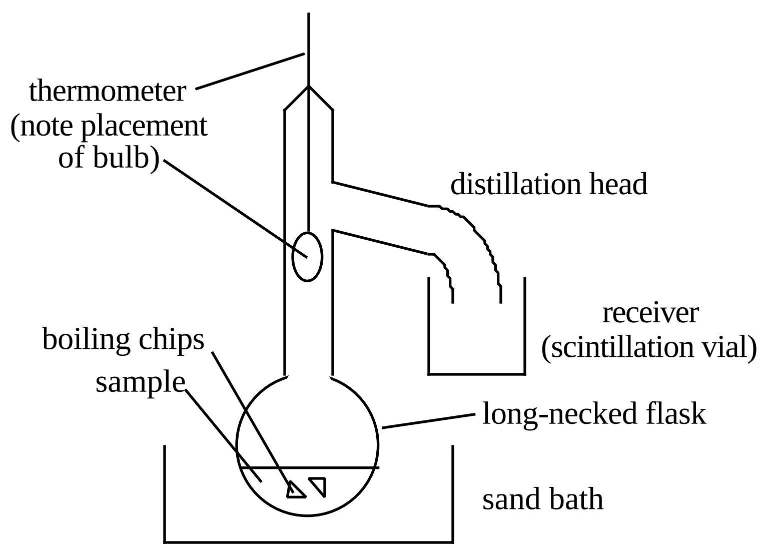
1. Here is some terminology on the subjects of this experiment.

   Boiling point: The temperature at which the vapor pressure of a substance equals the ambient pressure (that is, the pressure of the atmosphere around the sample).
   Refractive index: The ratio of the velocity of light in a vacuum to the velocity of light in a sample. Since it is a ratio of two velocities, it is dimensionless (that is, it has no units).
   Refractometer: Instrument used to measure the refractive index of a compound.
   Distillation: A process used to purify liquids in which a sample is evaporated at one location and condensed in another.
   Azeotrope: A mixture of liquids that distills at a constant temperature without changing composition. The best-known azeotrope is 95.5% ethanol and 4.5% water.

2. One factor that influences the boiling point is the pressure; the lower the pressure is, the lower the boiling point will be. Another factor is the presence of soluble impurities. Nonvolatile soluble impurities in a solvent increase the boiling point of the solvent.

   The refractive index changes with the wavelength of light used in the refractometer; most literature values of refractive indexes are taken using a wavelength emitted by sodium called the D line. The refractive index also changes with the temperature; many literature values (not all) are reported at 20˚ C. Finally, the presence of impurities can give a refractive index that differs from the literature value.

3. This is the setup we use for simple distillation:



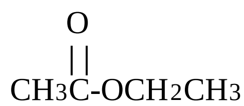
   The bottom of the flask is heated during the experiment, and the sample boils. The boiling chips or boiling stones provide nuclei for bubbles of vapor to form; without them, boiling is much less smooth. The hot vapors of the boiling liquid travel up the neck of the flask, heating the apparatus and finally the thermometer, until they reach the sidearm of the distillation head. The vapors finally condense in the sidearm, and the liquid drips into the receiver.

You must position the thermometer so that the entire bulb is just below the sidearm. This means that the bulb will be immersed in (and heated by) the hot vapors before the distillate starts to come over. This gives the most accurate measurement of the boiling point.

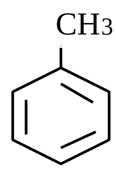
4. Here, once again, is the equation for the percent recovery from a distillation experiment (or any other experiment).

$$\text{Percent Recovery} = \frac{\text{weight of compound recovered}}{\text{weight of compound you started with}} \times 100\%$$

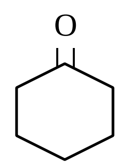
5. Here are the structures of the three possible unknowns for this experiment.



ethyl acetate



toluene



cyclohexanone

6. Safety considerations for this experiment:

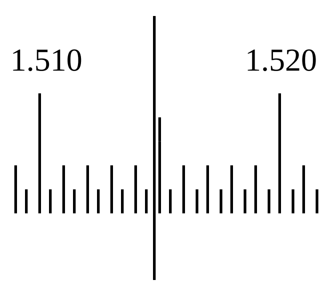
A sand bath, especially the top portion, can get very hot during the experiment, and you won't be able to tell by looking. Handle the sand bath with care. Also, a sand bath is an electrical apparatus; notify your instructor if its wires are frayed or exposed, and don't splash water on it.

You'll be using a mercury thermometer. These are fragile--handle them carefully, especially when you're installing them in the top of the distillation apparatus. If you break a thermometer and spill mercury, notify your instructor, who has the special spill kit needed to clean up a mercury spill.

All of the unknowns in the experiment are at least slightly toxic, and all are flammable. Avoid direct contact with these compounds.

Notes for topics that may be included in quizzes given after the experiment:

8. Here is a sample problem similar to one from an old quiz. What is the refractive index?



You'll find the answer on the next page. Important considerations: You must give four significant figures to the right of the decimal point, and interpolate between the two closest lines rather than reading the closest line only. The refractive index here is 1.5148.

To calculate a corrected refractive index, you'll need three pieces of information: the observed refractive index (RI) of your compound, the observed RI of water, and the literature RI of water (which is 1.3330). Here are the equations:

```
   Observed RI of water                  Observed RI of unknown
−  Literature RI of water      then          −   Correction
   ------------------------              ----------------------------
        Correction                       Corrected RI of unknown
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Sample problem: You determine the refractive index (RI) of your unknown using a refractometer, and it is 1.4414. Your observed RI of water is 1.3337. The literature RI of water is 1.3330. What is the corrected refractive index of your unknown?

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   1.3337                                 1.4414
−  1.3330                    then      −  0.0007
   ---------                              ---------
   0.0007 (the correction)                1.4407 (Corrected RI of unknown)
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Note: If your observed RI of water is higher than the literature value, the observed RI of your unknown will be higher than the corrected value, and vice versa. Both of the observed values will be higher (or they'll both be lower); there are no crossovers.

9. Here are some possible experimental errors and their consequences. Your quiz may include others.

    If you place the thermometer bulb too high, the vapors won't reach it (and heat it up) before they go into the sidearm to be collected, and your observed boiling point will be lower than it should be. If you place the thermometer bulb too low, vapors of impurities may reach it, giving a high reading for the boiling point range.

    Superheating most often happens when you leave out the boiling stones. Boiling of a superheated sample can be irregular and even explosive.

    If you try to distil in a closed system (this most often happens when you use a connector to attach your receiver), the pressure of the heated gases inside the apparatus will cause a rupture. Most often you'll launch your thermometer.

10. You've seen how to predict relative boiling points in the Organic Chemistry lecture course. You'll find more details in your lecture notes, but here are the general rules:

    First, identify the strongest intermolecular forces in your compound. Ionic bonds are the strongest, followed by hydrogen bonding, followed by dipole-dipole forces, followed by van der Waals forces. The stronger the forces, the higher the boiling point.

    If you have two compounds that have the same forces, consider molecular size. In general, larger molecules have higher boiling points than smaller ones do.

    If you have two molecules with the same functional group and the same molecular weight, consider the amount of branching. More branching means a lower boiling point.