Chem 224 Exp. #1 Simple Distillation FS2017

Separation of the components of a solution by distillation; boiling point and refractive index determination of the liquid component and melting point determination of the solid component.*

* The boiling point and refractive index (R.I.) are two useful physical properties of a liquid. They are used for identification purposes, and the R.I. is additionally used as a measure of purity of the sample being examined. The melting point (mp) is a physical property of a solid also used for identification purposes.

Reading Assignment: MTOL, pp. 72-85 (distillation theory), 49-52 (refractive index), 52-55 (melting point). Also read **CER TECH-700, 701, 702**

You are provided with a solution of an organic solid of low volatility in a solvent which can be distilled readily at a temperature not higher than 150 °C. Your task is to separate the components by distillation, record the boiling point (range) for the distillate and its refractive index, record the melting point (range) for the remaining solid, calculate the % error for MP, BP & RI in the recorded observations (by comparison with literature values), and comment on the discrepancies, if any.

1. Preheat the hot plate and Al block at a heat setting of ~130-145 °C while you assemble your glassware.

2. Put together a simple distillation set-up **(TECH 702, Fig. 7)**. Transfer the given solution to a 5-ml conical vial, filling it within 1/2" below the cap. Record the volume. Add your large magnetic stir bar (to regulate boiling and avoid bumping), and attach the Hickman still head and thermometer as shown. Regulate the boiling rate so that a smooth slow distillation is attained.

When distillate has accumulated in the Hickman still, using a pipet, transfer the accumulated first fraction to a 1-mL vial, cap it, and label it **(FORERUN)**. About ¼" of liquid in the vial is an adequate sample.

Continue the distillation of most of the remaining material and record the boiling range. Transfer this second fraction to a second vial, cap it and label it **(MAJOR FRACTION).**

There should be only 1/8" to ¼" of liquid remaining in the bottom of the flask. If the distillate fractions have all been collected and more still needs to be removed, disconnect the Hickman head and allow the vapors to evaporate directly into the mini hood.

Avoid excessive heating at this point, which may cause decomposition of the remaining solid. The last bit of liquid remaining is actually melted solid that will not evaporate, but may decompose if heated excessively. This will adversely affect the MP.

3. Let the distillation flask cool down, whereby the solute will crystallize out. Remove the solid with a microspatula to a filter paper. Let it dry out before checking its mp. You may help quicken the drying by patting the material with the filter paper to squeeze out excess solvent. Record the name and number of the MP apparatus used. Consult handout for MP determination instructions.

4. Determine the refractive indices of the two fractions on the Abbe refractometer **(TECH 702, Fig. 6**) and record your results. Record the temperature of the refractometer cooling water in order to correct the RI reading for temperature. Record the brand/type of refractometer used. Consult handout for operating directions.

5. List the boiling point and the refractive index for the solvent, and the mp of the solid as obtained from a Handbook listing such data. Compare to values in your prelab table to identify the solid & liquid in your unknown. (There will only be one of each). Calculate the % error in your reported mp, bp and corrected RI observations and comment on the outcome. Tabulate your results in your lab book. Show calculations.