Separation and Analysis of Three Unknown Liquids

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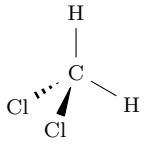
Lab Section 1A05

November 5, 2021

1. What is the identity of unknown D and how did you determine this?

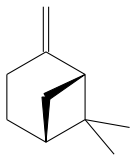


   – Unknown *D* is likely dichloromethane. By noting the temperature at which the first drop of liquid fell off the bulb of the thermometer during the distillation, we were able to determine the boiling point of the distillate as 32.5 °C. This most closely matches with the known boiling point of dichloromethane (39.5 °C to 40 °C).

2. Comment on the relative purity of unknown D and how you determined this.

   – Unknown *D* is likely not particularly pure since its experimentally determined boiling point is several degrees off from the actual. However, as impurities typically contribute to boiling point *elevation* (not depression), the difference in boiling point could also just be due to human/instrumentation error.

3. What is the identity of unknown E and how did you determine this?



   – Unknown E is likely (+)-β-pinene ((1R,5R)-6,6-dimethyl-2-methylidenebicyclo[3.1.1]heptane). Via polarimetry, we were able to determine the specific rotation of Unknown E as $[\alpha] = 18°$. This most closely matches with the known specific rotation of Unknown *E* (21°).

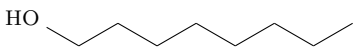
4. Comment on the relative purity of unknown E and how you determined this. Be sure to calculate your observed specific optical rotation and from that the enantiomeric excess (ee) as part of your answer.

   – In terms of the specific rotation, enantiomeric excess is given by the following formula.

$$\text{ee} = \frac{|\text{observed } \alpha|}{|\text{known } \alpha|} = \frac{18}{21} \approx 86\%$$

   Thus, the compound is fairly pure, but likely contaminated by some of another compound or some of its enantiomer.

5. What is the identity of unknown F and how did you determine this?



   – Unknown F is likely 1-octanol (octan-1-ol). By comparing the main gas chromatography peaks, we were able to determine the retention time of Unknown F as 2.391 min. This most closely matches with the known retention time of 1-octanol (2.415 min.

6. Comment on the relative purity of unknown F and how you determined this.

   – Unknown F is likely not entirely pure since there are many other smaller peaks in its gas chromatography spectrum. However, since the peaks are quite small relative to the 1-octanol peak, it is likely pretty pure.

7. For each of the two experiments this quarter (Separation of Unknown Solids and Separation of Unknown Liquids), a list of possible molecules was given. If these lists were not provided, how would this have changed how you determined the identity of any of the unknowns? How would you have modified the experiments to definitively prove the identities of A-F without this information?

- I could have compared the results of a number of characterization methods with a vast array of molecules, or I could have used more constructive methods. For instance, IR spectroscopy could have suggested possible functional groups, microwave spectroscopy could have determined bond lengths, NMR spectroscopy could have determined the number of each type of atom present, etc.

8. For each of the purification and analysis techniques that you have learned so far this quarter, list one strength and one drawback. These do not need to be in sentence form.

   (a) TLC.
   - Strength: Can identify impurities.
   - Drawback: Number of compounds that can be tested at once.

   (b) Melting point.
   - Strength: Can be measured to great precision.
   - Drawback: Takes a long time to measure.

   (c) IR spectroscopy.
   - Strength: Can identify the presence or lack thereof of specific functional groups.
   - Drawback: It does not tell you *how many* of each functional group you have.

   (d) Gravity filtration.
   - Strength: Only very simple materials required.
   - Drawback: With hot filtrations, you can lose a lot of your desired compound due to temperature differences.

   (e) Vacuum filtration.
   - Strength: Dries your precipitate even as it is separated.
   - Drawback: Loss of compound can occur from it splattering onto your gloves when you remove your hand from overtop.

   (f) Extraction.
   - Strength: Can separate solvents based on density.
   - Drawback: It is an art; it is easy to shake too little or too much.

   (g) Rotavap concentration.
   - Strength: Very fast evaporation.
   - Drawback: Loss can occur due to "bumping."

   (h) Recrystallization.
   - Strength: Allowing compounds to naturally regrow can lead to very high purities.
   - Drawback: Requires very careful manipulation (e.g., don't cool too fast, don't disturb too much).

   (i) GC.
   - Strength: Can analyze the purity of a substance.
   - Drawback: Cannot be performed by students in the lab.

   (j) Distillation.
   - Strength: Scalable — can be done in very large or very small quantities.
   - Drawback: Requires many resources (constant supply of cold water, constant supply of heat, etc.).

   (k) Column chromatography.
   - Strength: Can separate compounds by polarity in large volumes.
   - Drawback: Requires a lot of solvent and necessitates rotavaping afterwards.

   (l) Polarimetry.
   - Strength: Can differentiate between enantiomers.
   - Drawback: Susceptible to enantiomeric impurities (e.g., if the mixture is more racemic than we know, the value may be skewed even if its very high compound purity).