Lab 11: Investigation of a Chemical Bond by Infrared Spectroscopy

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Objectives

By the end of this laboratory, you should have developed the skills to do the following:

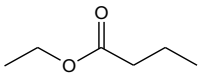
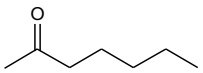
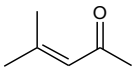
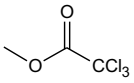
- Purify a compound via distillation.
- Run and interpret the IR of a reaction product.

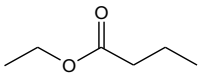
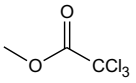
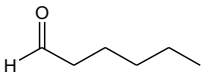
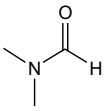
Recommended Resources

- Tutorial ~ Introduction to Distillation
  https://www.youtube.com/playlist?list=PL14A62ADC4C1C73D1
- Video ~ Organic Chemistry Lab Techniques – Distillation
  http://www.youtube.com/watch?v=FIIY53Ii_YI
- Video ~ Distillation - DanceChemistry
  https://youtu.be/1jU9jrqTz4M
- Video ~ Infra-Red Spectroscopy (How IR works)
  http://youtu.be/DDTIJgIh86E
- Video ~ Infrared Spectroscopy (How to Interpret an IR spectrum)
  http://www.youtube.com/watch?v=ItW6Mj2CQKc
- Website ~ Characteristic IR Absorption Frequencies of Organic Functional Groups
  http://www2.ups.edu/faculty/hanson/Spectroscopy/IR/IRfrequencies.html

Scenario

Hyacinth Pomander, from the Olfactory Factory, sent two sets of samples to your laboratory for analysis. One set was for the fragrance named "Musky Mix" and the other set was for the fragrance code named "Funky Mix" (see tables below).

Musky Mix				
A	 ethyl butanoate		**C**	 2-heptanone
B	 mesityl oxide		**D**	 methyl trichloroacetate

Funky Mix				
E	 ethyl butanoate		**G**	 methyl trichloroacetate
F	 hexanal		**H**	 dimethylformamide

Unfortunately, Hyacinth made two mistakes that will make your job considerably harder. First, she inadvertently used dirty containers to put all of the samples in. (The containers were previously used for a solution that consisted of both adiponitrile and ethyl acetate.) The second error was not using labels with a strong adhesive…all of the labels fell off! Fortunately, the samples were packed in separate boxes so at least you know which samples are from the Musky Mix and which samples are from the Funky Mix.

Background

Distillation is a method frequently used to purify liquid organic compounds. In distillation, compounds are separated based on differences in their boiling point. Simple distillation is used to separate compounds with significantly large differences in boiling points. (For mixtures of organic compounds that are very close in boiling point, fractional distillation must be used.)

Your research team will be assigned either the Funky Mix or the Musky Mix. Each team member will purify one sample via simple distillation, record its boiling point, and record its infrared (IR) spectrum. Together, you will determine the identity of each sample in the mix using its measured boiling point and the relative positions of the carbonyl stretching frequencies in the IR spectrum.

Lab Notebook Preparation

Before coming to lab, the following items must be in your lab notebook:

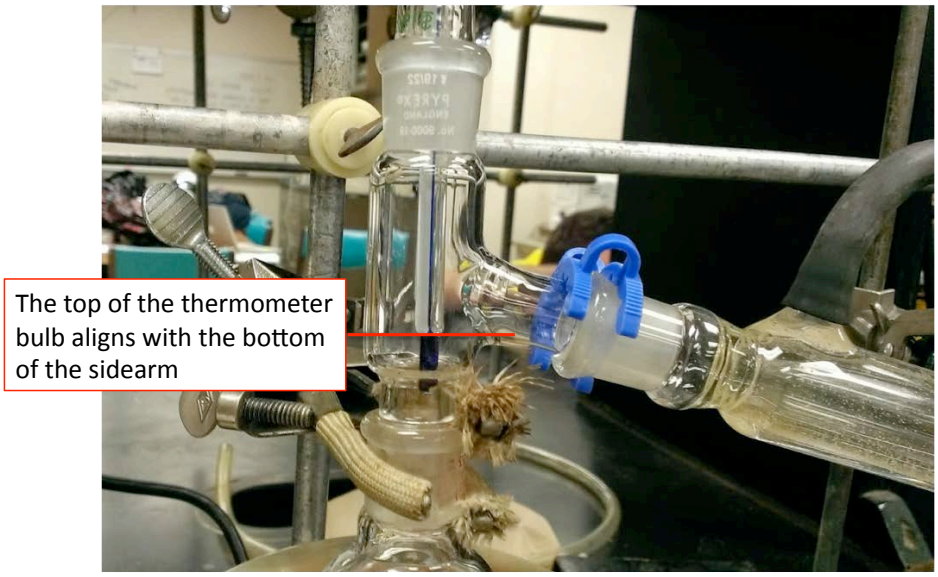
1. Title of experiment & Date the experiment is to be performed
2. Structures and boiling points of all possible unknowns
3. Structures and boiling point of ethyl acetate and adiponitrile
4. Important IR stretches for all possible unknowns
5. Important IR stretches for ethyl acetate and adiponitrile
6. Hazards of and appropriate precautions for the safe handling of unknown organic liquids
7. References

Safety Notes

- Assume that all unknowns are flammable and harmful by inhalation, ingestion, and skin absorption. Do not inhale their vapors and avoid contact with eyes, skin and clothing.
- When performing a distillation, ensure that you are not heating a closed system and that you do not heat the distillation to dryness.

Directions

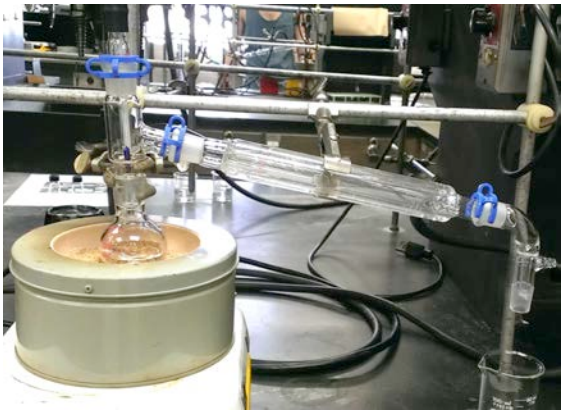
1. Place a magnetic stirrer into a 50 mL round bottom flask.
2. Add 20 mL of the assigned unknown into the round bottom flask.
3. Assemble an apparatus for simple distillation.
    a. Securely clamp the neck of the round-bottom flask to a support rod or ring stand.
    b. Place the round bottom flask in the heating mantle and the heating mantle on a stirring plate. (***Make sure the heating knob of the heating/stirring plate is off!***)
    c. Set the dial of the rheostat (the heat-control device) to the "0" or "off" position, and then plug the heating mantel into it. (***Never plug the heating mantel directly into an electrical outlet!***)
    d. Attach a three-way adapter to the top of the round bottom flask.
    e. Carefully slide the thermometer through the rubber stopper of the thermometer adapter (use glycerol or stopcock grease as a lubricant if necessary…do not force the thermometer!), and insert the thermometer adapter into the three-way adapter.
    f. Adjust the thermometer to the appropriate height (align the top of the thermometer bulb with the bottom of the sidearm as shown in the picture below).



    g. To the side arm of the three-way adapter, insert a condenser. (Support the condenser with an additional clamp.) Use a keck clip to hold it in place.

h. Attach the vacuum adapter to the end of the condenser. Use a keck clip to hold it in place.

i. Place a beaker under the tip of the vacuum adapter to use as a receiving unit.



j. Attach two black rubber tubes to the two outlets of the condenser. Connect the end of the tube attached to the lower outlet to the water source and place the end of the tube attached to the upper outlet in the drain.

k. Slowly turn on the water source, and adjust the rate until you have a slow but steady stream of water flowing through the condenser.

l. Start the stirrer, and check again to make sure that the heating knob of the heating/stirring plate is off.

4. Have your instructor check your apparatus before proceeding.

5. Perform a simple distillation to purify your unknown.

   a. Turn on the rheostat and adjust the heat so the liquid boils gently and the condensing vapors rise slowly.

   b. Once the vapors reach the thermometer, the temperature will rise very quickly. (If possible, adjust the heat so that the temperature rises at a rate of about 5 °C/min.)

   c. Record the temperature when the first drops of liquid begin to run through the condenser.

   d. Collect any lower boiling impurities (i.e., ethyl acetate). Note that the distillation temperature may drop and rise again as the lower boiling impurities are boiled off (you may need to adjust the rheostat again to achieve the proper heating rate).

   e. Once the temperature has stabilized at a temperature close to one of the of the samples, record the temperature and continue to collect the first few drops (which may still have residual ethyl acetate).

   f. Change the receiver flask and begin collection of the pure sample in a clean, dry beaker.

   g. Monitor the temperature frequently, and note any changes. If you see a significant rise in temperature, change the receiver flask.

   h. Stop sample collection when 1-3 mL of liquid remains in the round bottom flask or as soon as the temperature begins to rise again. (It is unsafe to allow a distillation to run to dryness!)

6. Determine and record the volumes of the liquids collected in each beaker. Once your apparatus has cooled to room temperature, record the volume of the liquid remaining in the round-bottom flask, and then carefully disassemble the apparatus.
7. If your boiling point does not match the boiling point of any of the possible unknowns, repeat your distillation using your purified unknown. (Note: A digital thermometer may be more successful at achieving an accurate boiling point.)
8. Analyze your sample using the IR Spectrometer.
    a. Refer to the handout "Appendix C: Instructions for Using the IR Spectrometer" for directions on how to use the instrument.
    b. Note the wavenumbers (cm^{-1}) of the major IR peaks.
    c. Determine the wavenumber (cm^{-1}) of your compound's carbonyl (C=O) band from its spectrum and record this number. (Generally, the carbonyl band will be the only strong band between 1780 cm^{-1} and 1630 cm^{-1}.)
9. Store some of the remaining pure sample in a capped vial, clearly label it, and keep it in your drawer until you have completed the analysis.
10. Share your results with the rest of your team to determine the identity of each sample in your assigned mix. Your team may choose to redistill some of the samples if impurities are present or if the results do not make sense. If desired, and with your instructor's permission, you may confirm your results using NMR.

Reporting Your Results

Write your report according to the guidelines described in "Topic 4: Writing an Organic Chemistry Lab Report". Work with your group on this report.

References & Additional Resources

1. Lehman, J. W. *Operational Organic Chemistry: A Problem-Solving Approach to the Laboratory Course*, 3rd ed.; Prentice Hall: Upper Saddle River, NJ, 1999; pp 102-108.