Identifying Unknowns by IR Spectroscopy

Purposes of this lab:

- Learn to use the IR spectrometer.
- Learn to identify an unknown compound by its IR spectrum.
- Practice recognizing the functional groups of IR spectra.

Introduction:

Infrared spectroscopy is an extremely useful tool for identifying and characterizing organic compounds.

In this lab, you be trained to use the IR spectrometer, and obtain spectra of two unknown compounds. You will then identify them from a list of possible compounds (given below).

Since only one person can use the spectrometer at a time, you will also have a set of printed spectra to match up with others of the same functional group.

Procedures:

Before you come to lab:

- Print out a copy of this lab and read through the introduction and procedures. Put it in your binder and bring it to lab.
- Print out a copy of "Characterizing Compounds by Infrared Spectroscopy (IR)" (found on the course website), and put it in your binder as well. Read it carefully.
- Write an introduction in your lab notebook explaining the purpose of the lab in your own words.
- Complete the pre-lab questions on-line before the deadline.

During lab:

Part 1: Identifying an Unknown by IR

- Copy the table at the end of this lab into your lab notebook. Fill in the functional group and the identifying bands for each compound (including frequencies). The first one has been filled out for you to show you how it should look.
- Choose one of the unknown compounds on my desk. You will then be grouped with four other students. All four of you will work together on all four unknowns. Write the letters of the unkowns in your notebook.
- Each group will have a turn at the spectrometer. As your turn comes, bring your compounds and a pipet for each to the IR spectrometer. Take a spectrum of both compounds (each student should take one spectrum). Follow the instructions given in "Characterizing a Sample by IR Spectroscopy" carefully.

- Print a copy of all four spectra for each student.

- Working together, identify and label the important functional group bands on each spectrum. Decide which of the compounds in the table you think it is. Make sure that you can eliminate all others based on bands that are present or absent.

Part 2: Matching printed spectra

- Obtain a packet of spectra from the instructor. Lay out each of the labeled spectra, with room for 4 more underneath. Match up the rest of the spectra by comparing them to the labeled ones. There are 4 of each (except alkynes, which only have 3). You do not need to write anything about these spectra in your lab notebook.

After lab:

- Write a conclusion in your lab notebook, stating what unknown compounds you believe you have and what evidence supports this.

- Answer the questions below in your lab notebook.

- Turn in the yellow carbon copies of your lab notebook and your labeled spectra on the day they are due.

<u>Questions</u>

1) Why is it necessary to have the computer collect a background as part of the procedure for taking a spectrum? (1 pt)

2) When the only information you have is an IR spectrum, why is it necessary to have a list of compounds to chose from? (1 pt)

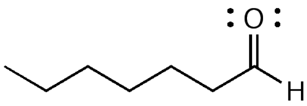
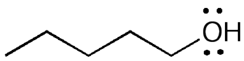
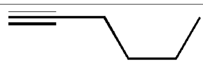
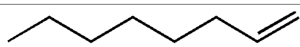
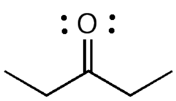
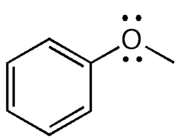
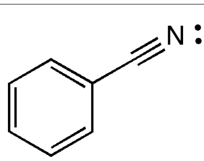
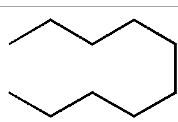
3) The absorption for carbon dioxide appears around 2300 cm^{-1}. Spectra often contain this band. Do any of your spectra show this absorption? Why won't it always be taken out by subtracting the background spectrum? (2 pts)

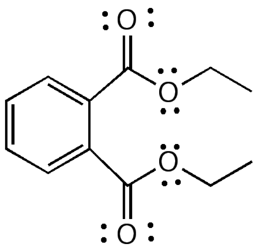
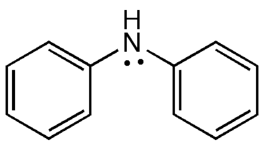
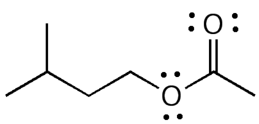
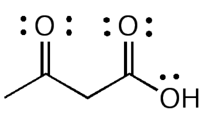
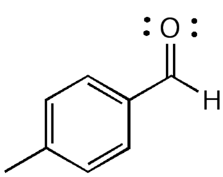
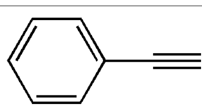
4) C=O and C=C bands are in the same region but are not very difficult to tell apart. Explain how you could distinguish between 1-octene and 3-pentanone by IR spectroscopy. (2 pts)

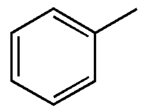
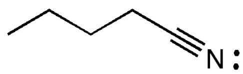
5) How are the O-H bands of an 1-hexanol and levulinic acid different? What else can you use to tell alcohols and acids apart? (2 pts)

6) Ketones and esters have very similar spectra because both have C=O bands in their spectra. What band should you look for on the spectrum of isopentylacetate that a spectrum of 3-pentanone won't have? (1 pt)

7) How could you tell the difference between 1-hexanol, $CH_3(CH_2)_5OH$, and 1-hexanamine, $CH_3(CH_2)_5NH_2$? How are the bands between 3400 and 3200 cm^{-1} different? What other bands would be different between these two compounds? (2 pts)

compound	functional group	identifying bands
 1-heptaldehyde	aldehyde	C=O (1800-1650 cm^{-1}) C-H on C=O (about 3300 cm^{-1})
 1-hexanol		
 1-hexyne		
 1-octene		
 3-pentanone		
 anisole		
 benzonitrile		
 decane		

 dibutyl ether		
 diethyl phthalate		
 diphenylamine		
 isopentyl acetate		
 levulinic acid		
 p-tolualdehyde		
 phenylacetylene		

 toluene		
 valeronitrile		