

## 12AL Experiment 6: Infrared Spectroscopy

### Safety:

This is mainly a “dry lab” with the exception of the unknowns to be run on the IR spectrometer. Please clean IR trough with isopropyl alcohol BEFORE & AFTER you run a sample. Throw away all trash. Make sure you have recorded your unknown numbers appropriately on your printed IR spectrums. Return unknown vials to instructor – do not keep in your drawer!

### Background:

Infrared Spectroscopy is a very powerful tool to help uncover the identity of an unknown and/or to check the purity of a compound.

Atoms and chemical bonds are not stagnant – they are constantly in motion, vibrating at unique frequencies. There are several motions that chemical bonds undergo – stretching, bending, wagging – to name a few. The most basic description of how an IR spectrometer works is the following: A compound is put into the IR and infrared light is passed through the sample. When a specific frequency of the infrared light matches the unique frequency of a chemical bond, the chemical bond “over-stretches,” and this exaggerated pulse is recorded by the machine on a spectrum. Every unique chemical bond has a specific frequency and shape that the spectrometer records, and thus, you are able to determine the types of bonds and functional groups within a compound. While this information may not give you the exact structure of the molecule, it is still very powerful information as you can determine all functional groups present. It is your job, to memorize all chemical bond “wavenumbers” as you will be using IR spectroscopy almost everyday.

The IR spectrometer measures bond frequencies in terms of wavenumbers. Wavenumbers are simply frequencies in units of  $\text{cm}^{-1}$  instead of traditional frequency units of  $\text{Hz}$  ( $\text{s}^{-1}$ ). You will be looking up all the wavenumbers for chemical bonds you most commonly use as part of your prelab – it is important that you begin to memorize them as well as look up their shapes in examples throughout your lecture textbook, lab manual, and internet. As an organic chemistry student, you are required to know your IR bond wavenumbers. A very useful website for viewing IR spectrums of millions of organic compounds as well as NMR spectrums and other information can be found at: [http://sdb.srioddb.aist.go.jp/sdb/cgi-bin/cre\\_index.cgi](http://sdb.srioddb.aist.go.jp/sdb/cgi-bin/cre_index.cgi).

To help determine the structure of a compound depicted in an IR spectrum, you can also calculate the degrees of unsaturation from its molecular formula. The degrees of unsaturation can give you some useful structural information and may serve as a guide to the IR spectrum:

1° can be 1 Double Bond OR 1 Ring

2° can be 1 Triple Bond OR 2 Double Bonds OR etc..

3° can be 1 Triple Bond AND 1 Double Bond, OR etc...

4° is commonly a Benzene structure (1 Ring with 3 DB)

Notice that the degrees of unsaturation often gives information about double and triple bonds – a good method to practice whenever you calculate 1-4° is to check for the C=C wavenumber or the C≡C wavenumber. If either a DB or TB is present, you also need to check the placement of it – is it an internal DB/TB or a terminal DB/TB? Recall, terminal DB/TB have an additional wavenumber present which would be noticed by the presence of C=CH<sub>2</sub> or C≡CH.

Degrees of unsaturation are calculated by comparing the hydrogens present in your given molecular formula to the hydrogens that would be present in an alkane with the same carbon count. You will go into more detail in your lecture course, but here is a quick review of the basics behind calculating the degrees...

Alkanes are saturated with hydrogen atoms. Carbon makes four bonds and in an alkane, carbon has all single bonds, thus being saturated with hydrogen atoms. When, for example, a double bond or ring is introduced into the structure, two hydrogen atoms are lost, giving 1° of unsaturation. When, for example, a triple bond is introduced into the structure, four hydrogen atoms are lost, giving 2° of unsaturation. Below is the equation you need to use to calculate the degrees of unsaturation. Make sure you become very comfortable with it!

$$\text{Degrees of Unsaturation} = \frac{(\text{\#Hs in an Alkane w/ same \#C}) - (\text{\#Hs present in given formula})}{2}$$

WHERE... (#Hs in an Alkane w/ same #C) is found by recalling the following....

Alkanes have 2n+2 H atoms where n = #C atoms

AND...

(#Hs present in given formula) is affected by heteroatoms in the following way...

Halogens: each halogen adds 1 to #Hs present in given formula

Oxygen: does not change #Hs present in given formula

Nitrogen: each nitrogen subtracts 1 from #Hs present in given formula

For example, CH<sub>3</sub>Cl has  $\frac{(4) - (3 + 1)}{2} = 0^\circ$  (all single bonds)

C<sub>4</sub>H<sub>8</sub> has  $\frac{(10) - (8)}{2} = 1^\circ$  (could be cyclobutane, 1-butene, or 2-butene)

C<sub>4</sub>H<sub>8</sub>O has  $\frac{(10) - (8)}{2} = 1^\circ$  (may have a C=C or a C=CH<sub>2</sub> or a C=O or could even be cyclobutanol)

## Objective:

Day 1: To determine the structure of the following organic compounds given their molecular formula and IR spectrum. To learn how to calculate degrees of unsaturation given a molecular formula as this information helps in IR analysis. To label ALL bonds in a determined structure on its IR spectrum. Day 2: To run two unknowns on the IR, print out their spectrums, and analyze the spectrums to determine their structures as in Day 1.

## Procedure:

1. For each spectrum below perform the following: (ALL WORK MUST BE SHOWN)

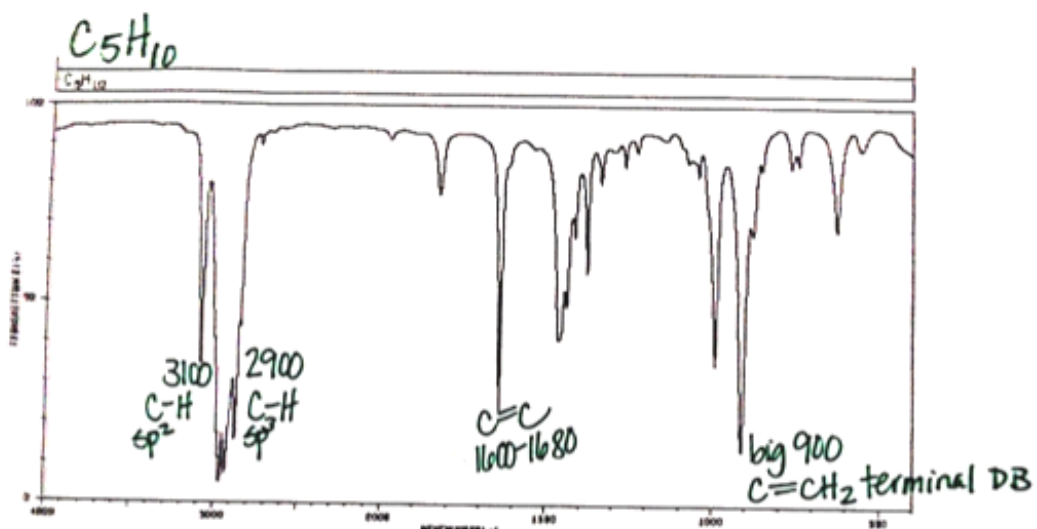
- A. Calculate the degrees of unsaturation. Think about what this information tells you.
- B. Label the IR as shown in the example. First label the obvious IR stretches. Once you have deduced the functional groups present and the structure, make sure ALL BONDS in your structure are labeled on the IR spectrum with their corresponding wavenumbers.
- C. Make sure your final structure is drawn on the spectrum & circle it.

\*You will be analyzing IR spectrums in almost every experiment you perform in your year of organic chemistry. Please get familiar with the instructions above (A, B, C) as you are required to analyze the spectrum in this fashion everytime to prove your work.

\*Remember, this is individual work – if you rely on your neighbor to do the work or start it for you, then you will find great difficulty in IR quizzes & future experiments.

2. When you have completed the spectrums below, turn it in to your instructor. They will return the packet to you on Day 2 with any incorrect spectrums marked – YOU ARE REQUIRED TO FIX ANY INCORRECT SPECTRUMS ON DAY 2 *BEFORE* YOU BEGIN YOUR UNKNOWNNS.

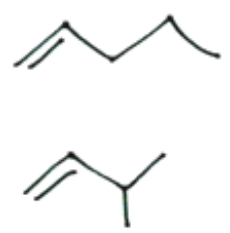
3. Run two separate unknowns on the IR; make sure to write down your unknown numbers on the spectrums you will obtain!; print their spectrums, analyze their spectrums as learned, determine their structure, and turn final work into instructor to be graded.



deg. of unsaturation C<sub>5</sub>H<sub>10</sub>: Alkane w/ 5 Cs has  $(2 \times 5) + 2 = 12$  Hs  
 so...  $\frac{12 - 10}{2} = \frac{2}{2} = 1^{\circ}$  **1 DB**  
 ↳ RING

possible structure(s) that match IR:

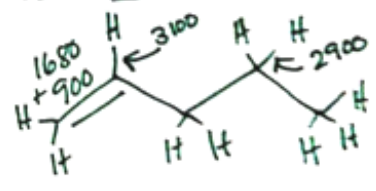
terminal alkenes that contain alkane sp<sup>3</sup> C-H bonds too.



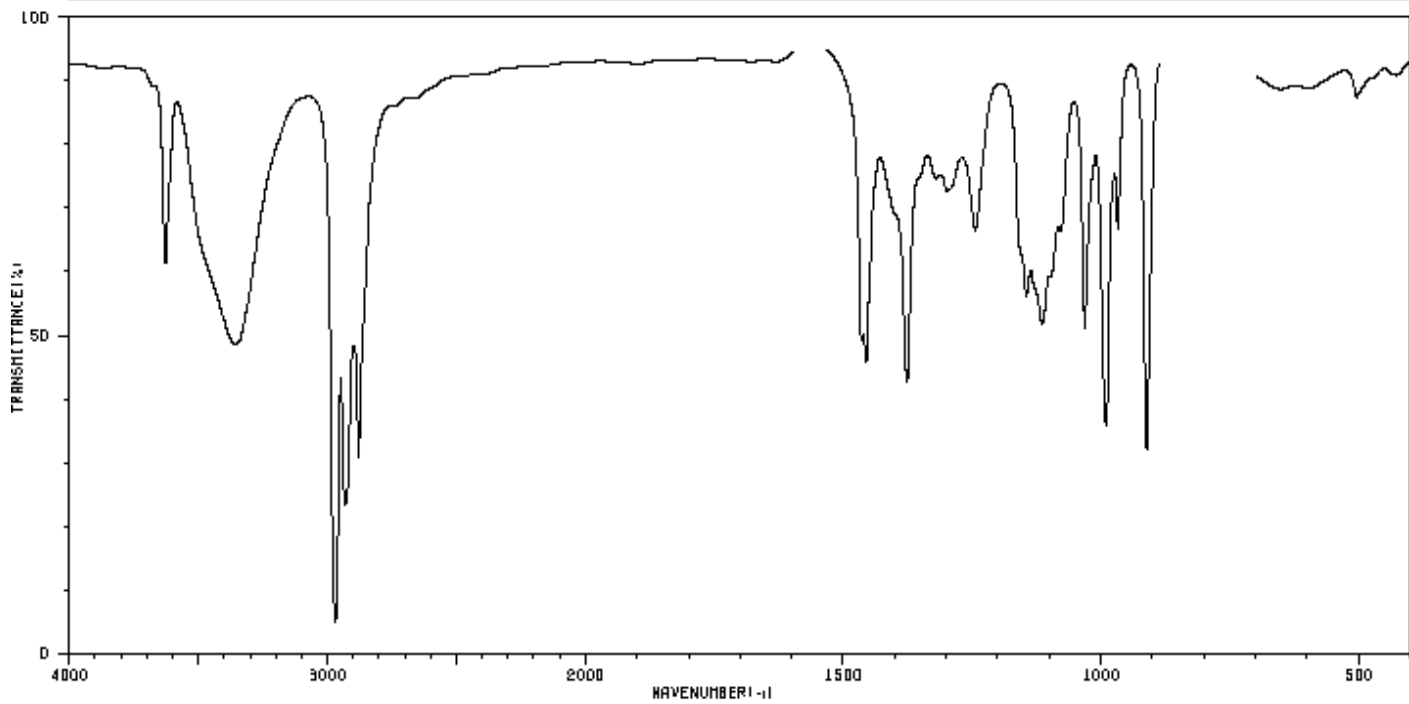
1-pentene

3-methyl-1-butene

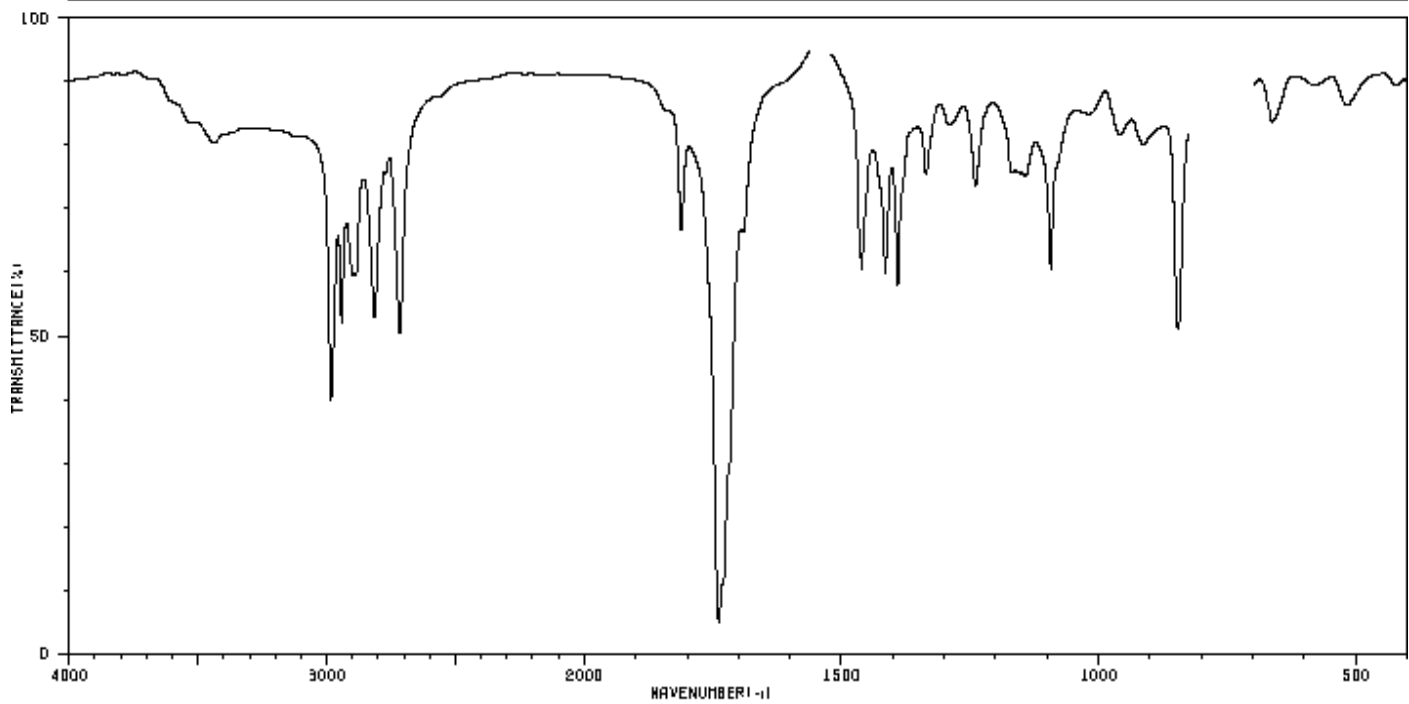
etc...

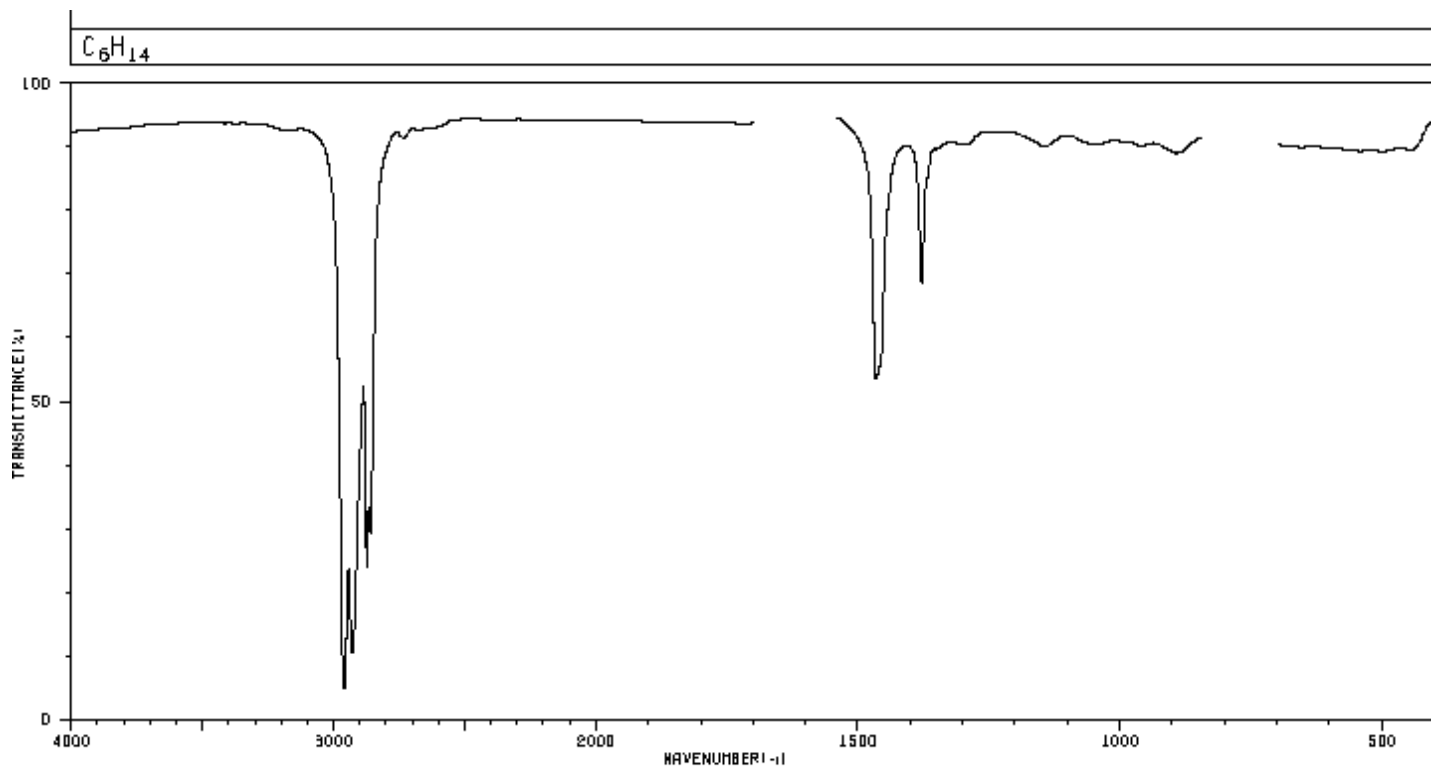
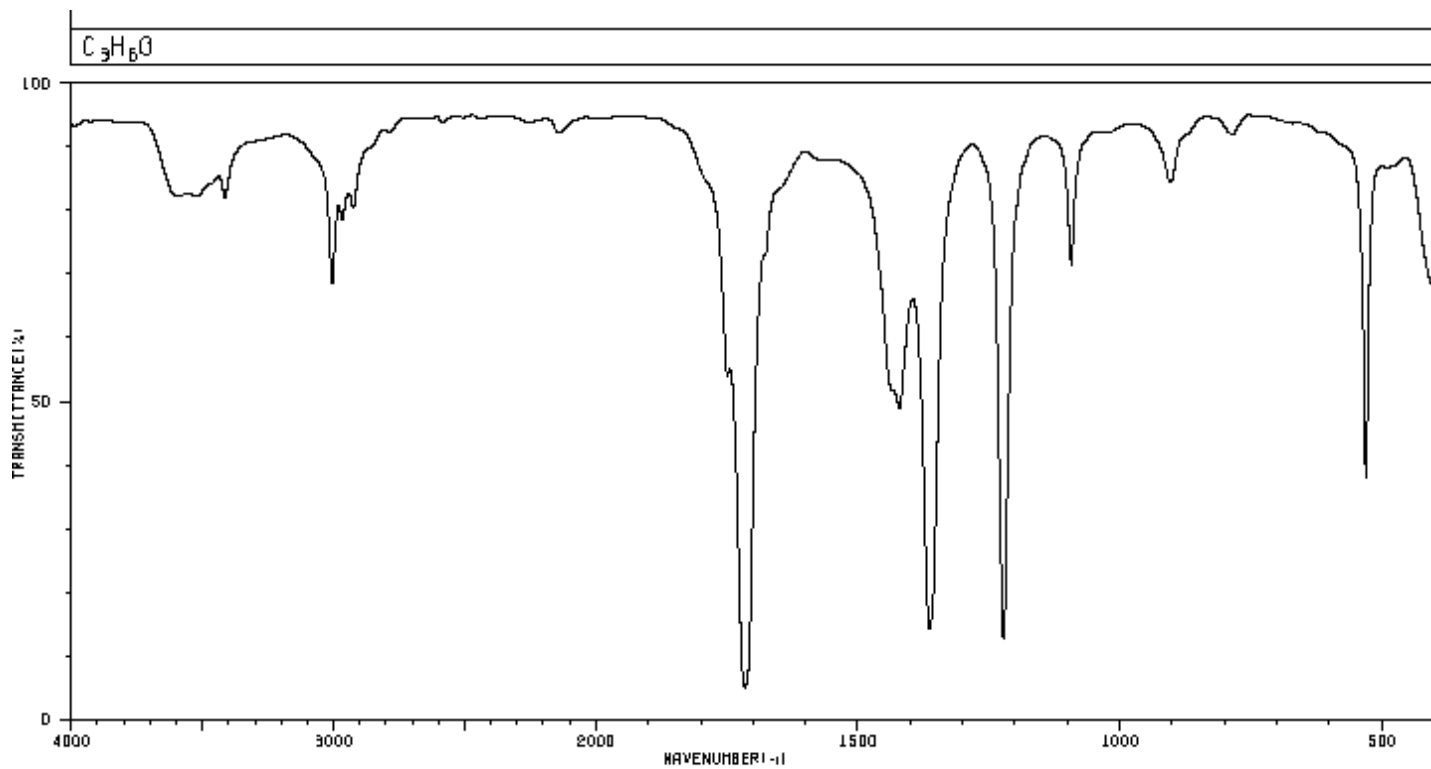


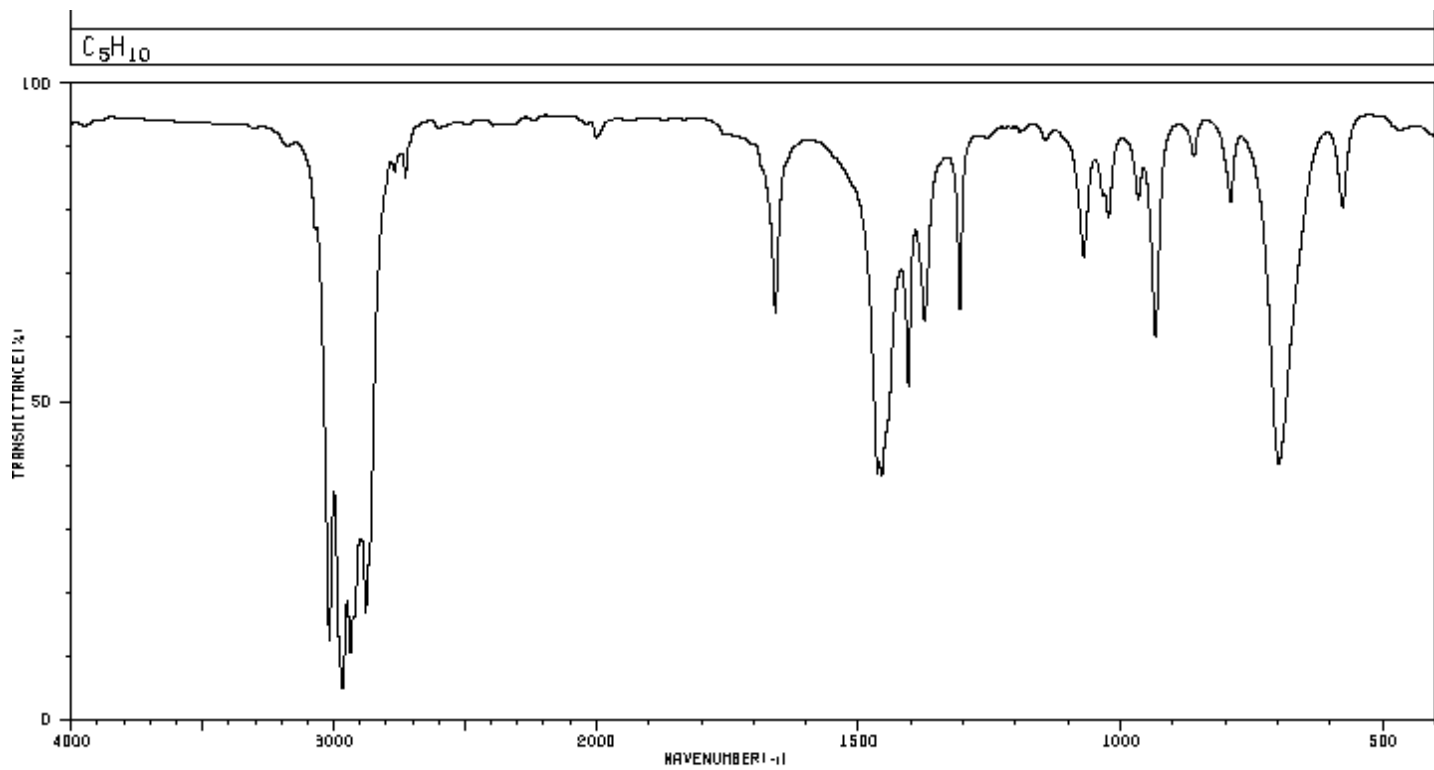
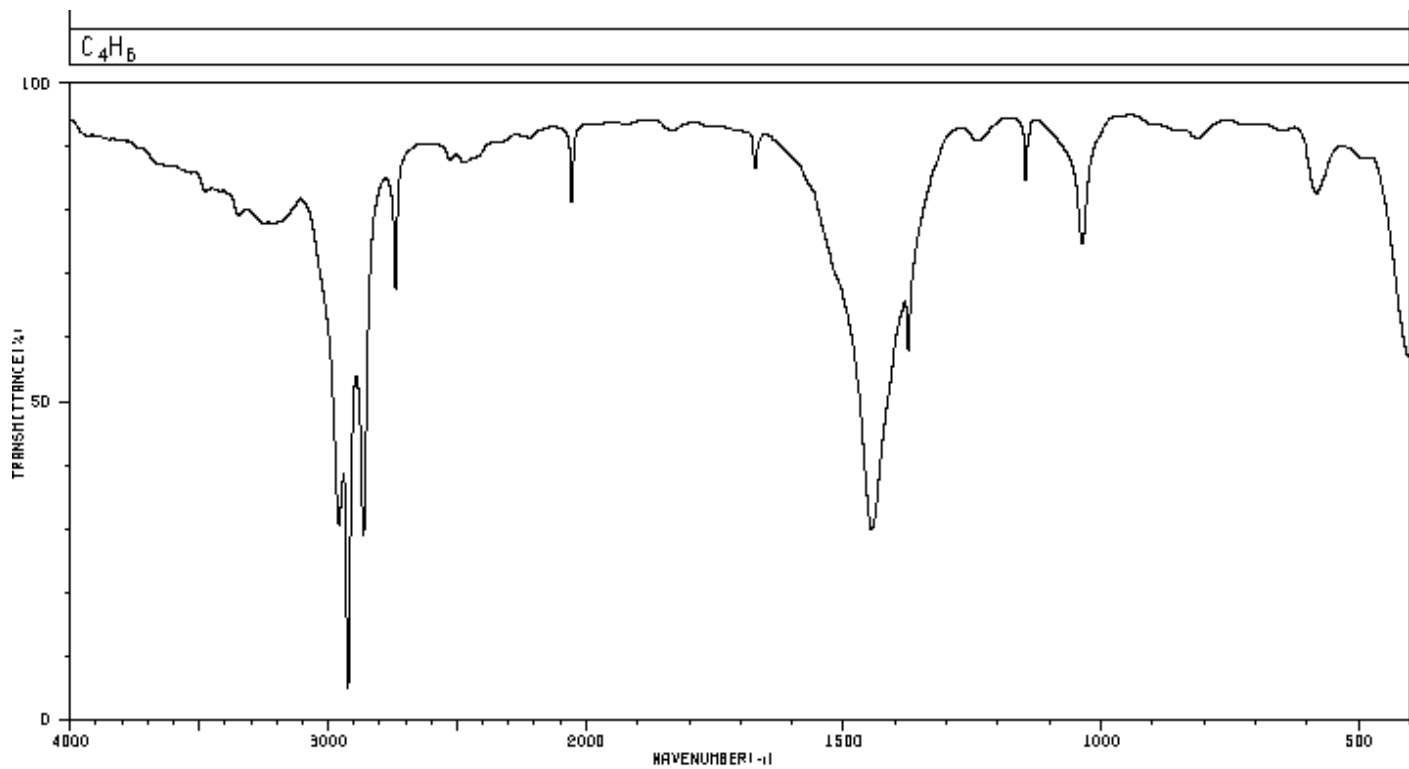
$C_4H_{10}O$



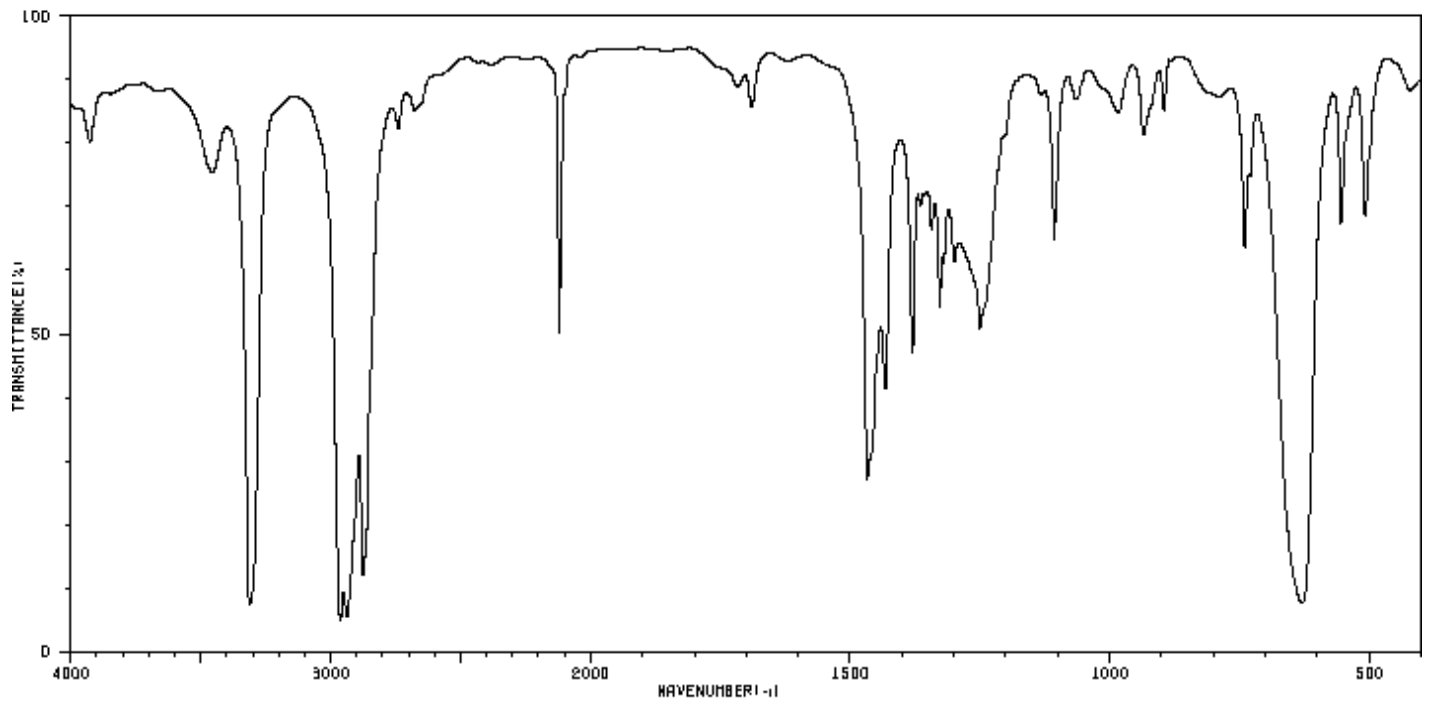
$C_3H_6O$



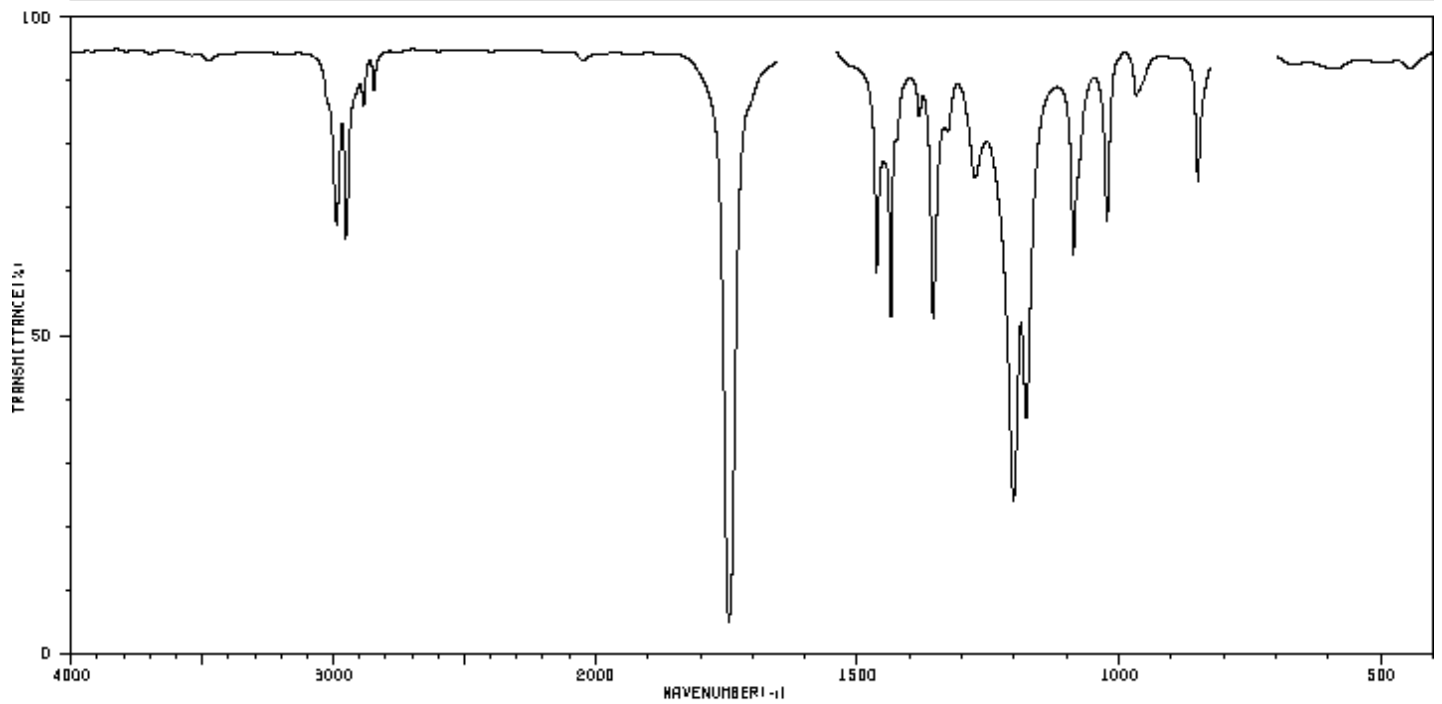




$C_6H_{10}$



$C_4H_8O_2$





## 12AL Prelab Experiment 6: Infrared Spectroscopy

1. Look up the bond wavenumbers for each of the following in your lecture textbook - be sure to read thru the IR chapter for specifics like sp<sup>2</sup> vs sp<sup>3</sup> carbon-hydrogen bonds!

C-H (C is sp<sup>3</sup>)

C-H (C is sp<sup>2</sup>)

C-H (C is sp)

C=C (all alkenes)

C=CH<sub>2</sub> (careful this is not the same as the previous; this is a terminal alkene)

C=C (aromatic alkene like benzene)

C≡C (all alkynes)

C≡CH (careful this is not the same as the previous; this is a terminal alkyne)

O-H

O-H (of COOH)

C=O (approximate of all C=O containing molecules)

C-O

2. How many degrees of unsaturation are there in...

An alkene?

Ester?

An alkyne?

Ketone?

Benzene?

Cycloalkane?

Carboxylic Acid?

Alkane?

3. Give three different structures each with 5°, all that have the most common 4° structure.

## 12AL Postlab Experiment 6: Infrared Spectroscopy

1. Indicate which one of the following 6 compounds corresponds to each of the IR spectra below. Once matched, LABEL ALL BONDS present in the structure ON THE IR SPECTRUM just like the lab experiment.

