Infrared Spectroscopy (IR)

OBJECTIVE

1 To identify various functional groups in Organic Chemistry

2 To learn of the important role of infrared spectroscopy in the study of structure of organic compounds

3 To develop skill in the recognition of characteristic absorption bands

4 To identify a compound by an investigation of its infrared spectrum

INTRODUCTION

During the course of this year we will study several different classes of compounds including alcohols, alkenes, and ketones. Each of these classes is distinguished by the presence of a "Functional Group"[1] in the molecule. For example, all alcohols contain an 'O-H' group attached to an sp^3 hybridized carbon atom. Alkenes contain a carbon-carbon double bond (C=C), and ketones contain a carbon-oxygen double bond (C=O).

As you will learn, IR is also referenced as a Functional Group detector.

The quickest and easiest way to determine the presence of one of these "Functional Groups" is to take the IR spectrum of the compound. The technique is simple and can often provide a definitive answer in less than five minutes. Evidence provided by IR is widely respected. It is commonly used in judicial proceedings as much as fingerprints are used. In fact, the IR of a pure compound bears the same relationship to that compound as fingerprints do to an individual.

Be sure to read the section on infrared spectroscopy (IR) of your text before doing your first IR to understand the technique and information the spectrum provides.

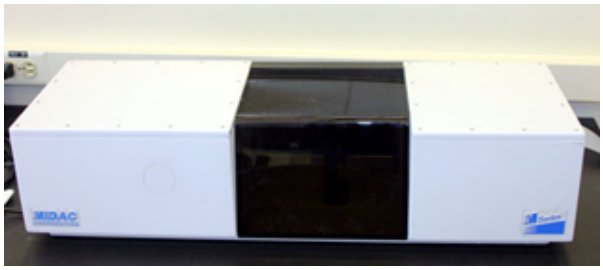


Figure 1: Bruker Alpha I FT IR Instrument

An IR instrument consists of an IR light source, a sample holder, a means of selecting individual wavelengths or frequencies of the light, some means of detecting the amount of incident light that the sample absorbs, and a device for plotting the amount of light absorbed as a function of wavelength or frequency. This plot is referred to as the 'IR Spectrum.'

[1] http://en.wikipedia.org/wiki/Functional_group

Sampling

IR spectra can be determined for solids, liquids, or gases.

Solids and liquid samples are placed directly on the diamond crystal plate. For a liquid sample, use a glass pipet or pipettor to place a small drop of the sample on the plate to cover the diamond crystal. For a solid, apply enough powder to coat the crystal. Position the pressure arm over the sample and apply pressure to the sample.

After the IR has been taken of the unknown, clean the crystal by applying EtOH to a Kimwipe then blot the crystal and wipe it clean. If the sample was solid, also clean the anvil tip of the pressure arm.

IR gas analysis is a common analytical tool for those involved in studies of atmospheric pollution. The only draw-back is that it is very expensive and delicate cells are needed.

Reference Spectra

The Infrared spectra of thousands of compounds have been determined and compiled by several different companies. Two of the most popular collections are the Sadtler Index of IR Spectra and the Aldrich Library of Infra-red Spectra

Both collections are easily accessible in 'hard copy' form in most major university libraries. They are also available in computer readable format for rapid searching and spectrum matching. All modern FT-IR spectrometers are controlled by computers. The operating system often has the capability of searching one or more databases of spectra and finding the spectra that most closely match the spectrum that was just run.

It is always a good idea to compare your IR spectrum with an authentic spectrum of the material you think you have.

ALL IR Spectra in this manual are reproduced with permission from: The Aldrich Library of FT-IR Spectra, Edition 1, Charles J. Pouchert, Volume 1, 1985.

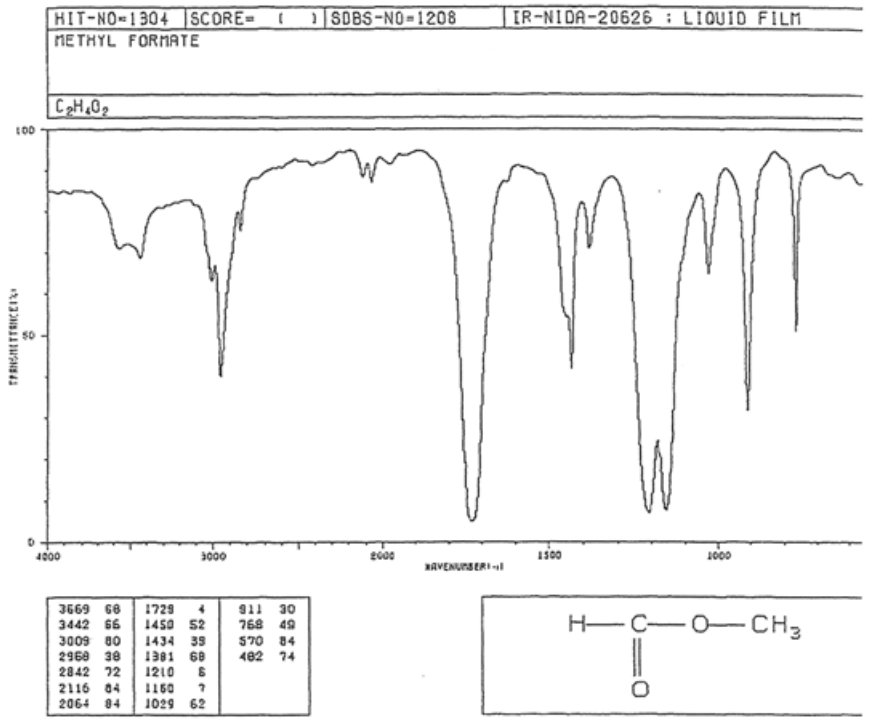


Figure 2: Methyl formate - An ester

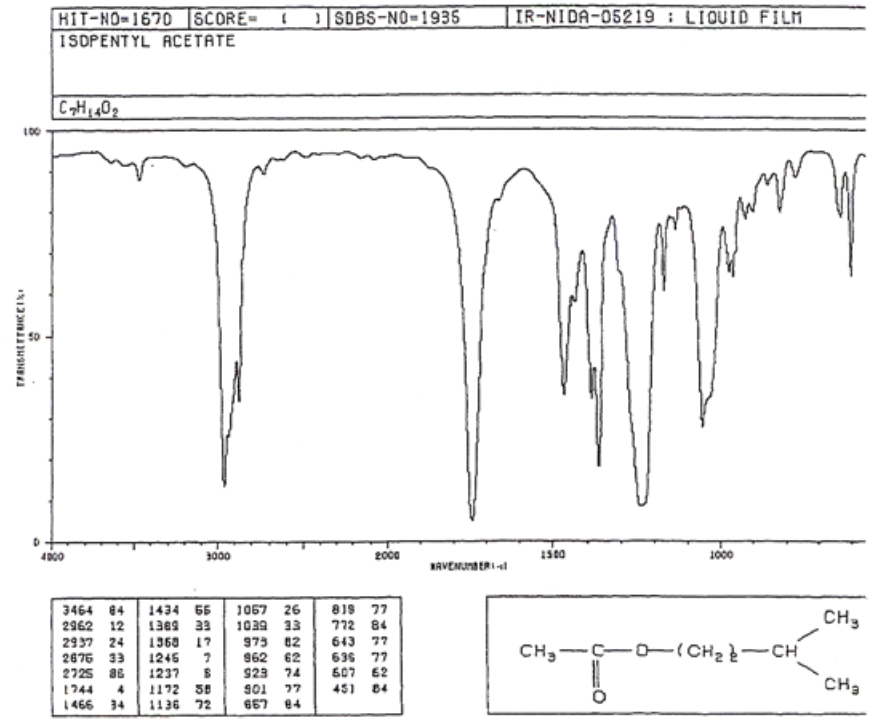


Figure 3: Isopentyl acetate - An ester

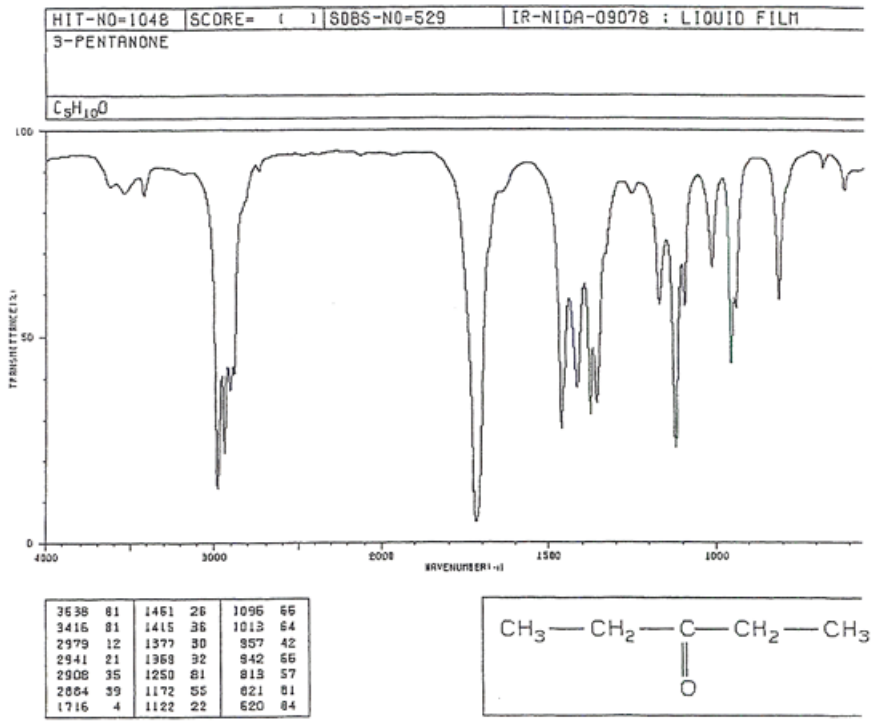


Figure 4: 3-Pentanone - A ketone

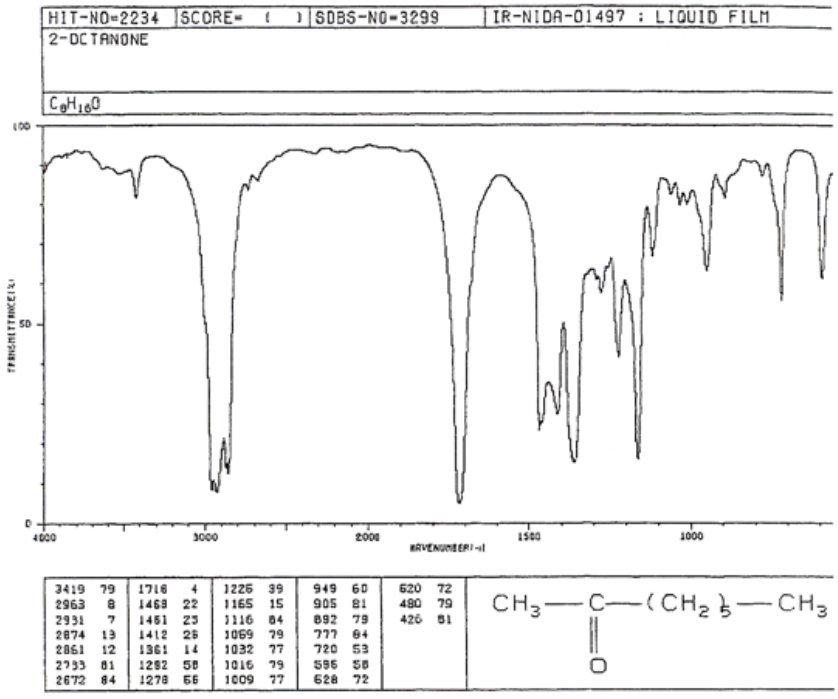
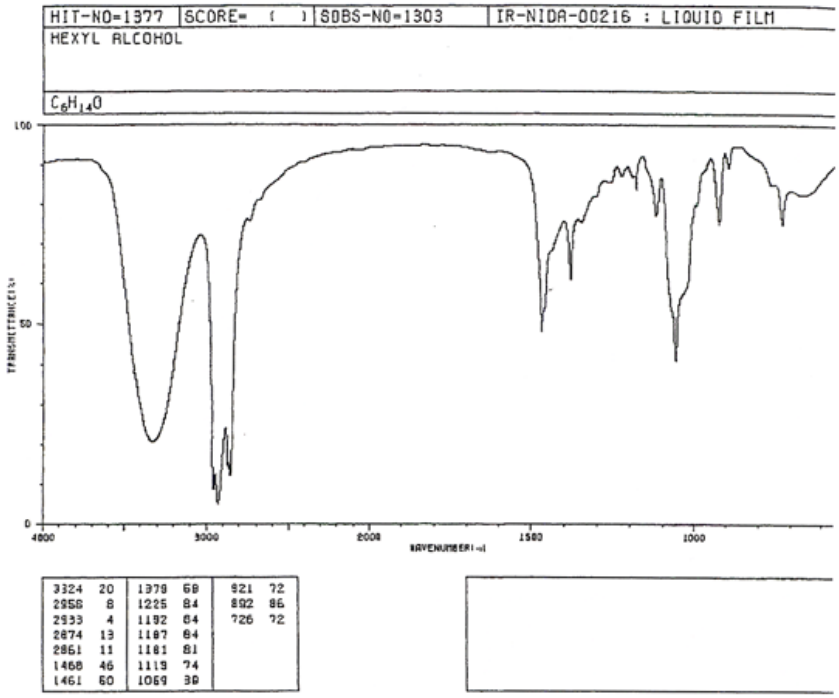


Figure 5: 2-Octanone - A ketone



$CH_3CH_2CH_2CH_2CH_2CH_2OH$

Figure 6: 1-Hexanol - An alcohol

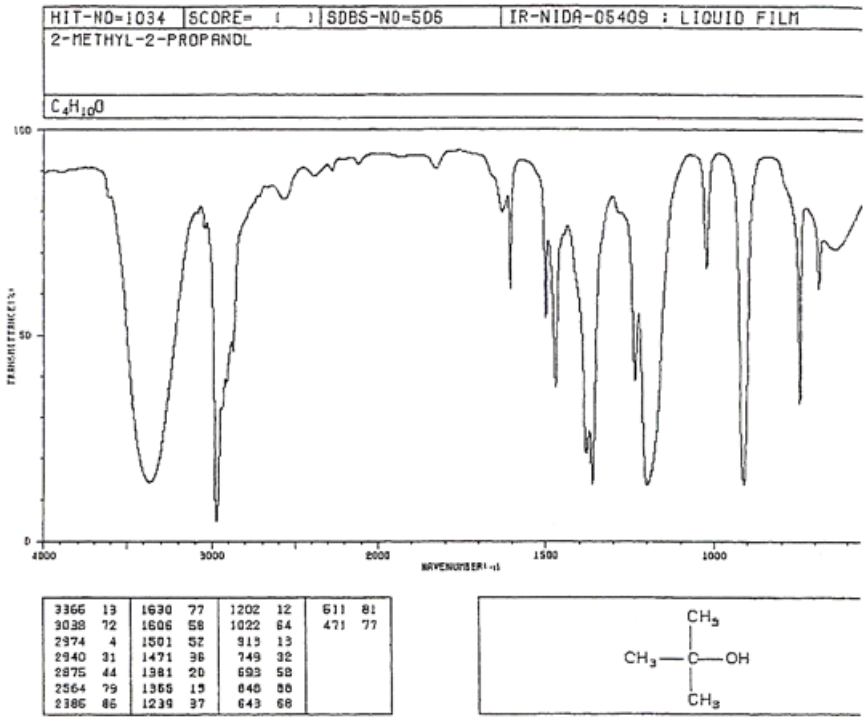


Figure 7: 2-methyl-2-propanol

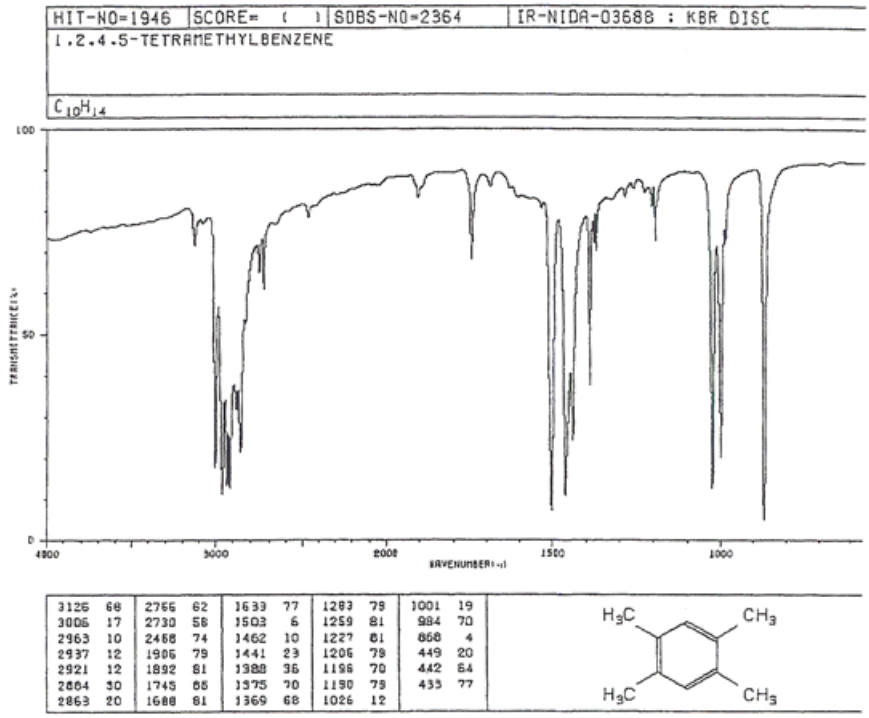


Figure 8: 1,2,4,5-Tetramethylbenzene - An aromatic

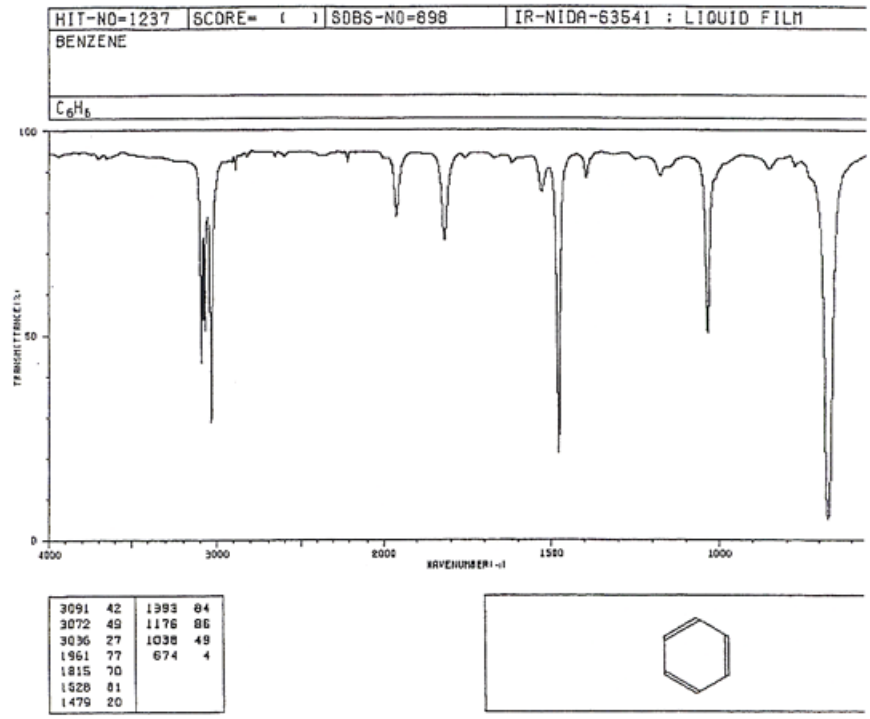


Figure 9: Benzene - An aromatic

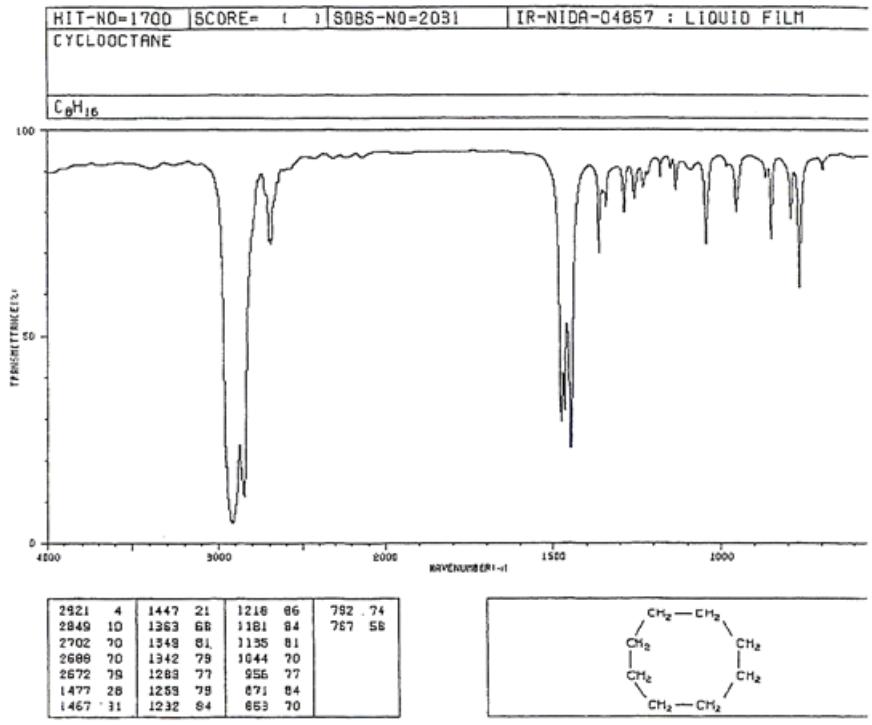
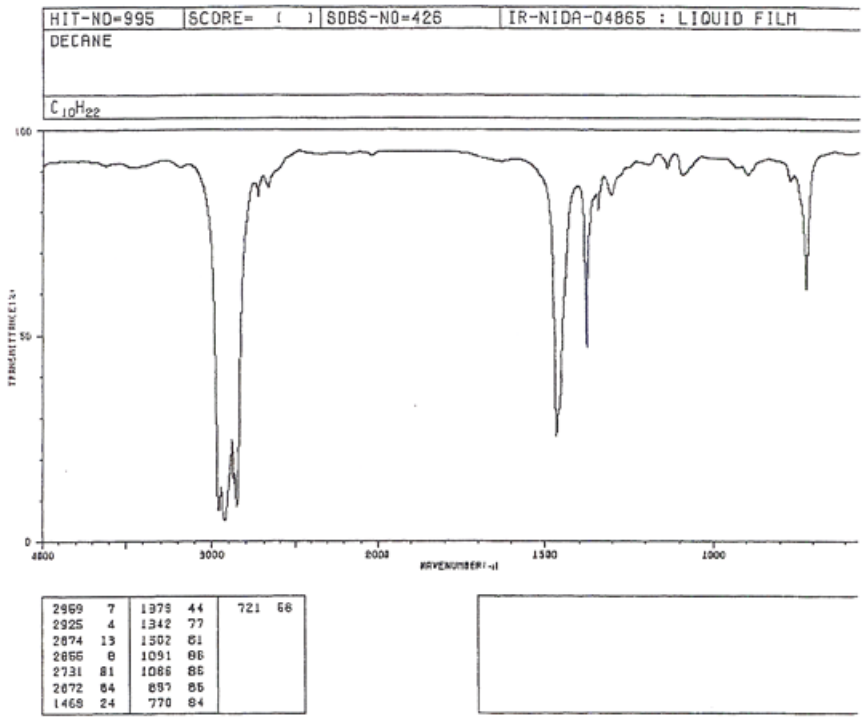


Figure 10: Cyclooctane An alkane



$CH_3CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_3$

Figure 11: Decane - An alkane

Functional Groups

We have seen that IR is really a **"Functional Group Detector."** As such, it will be a valuable tool throughout this year. You should become accustomed to examining an IR spectrum and looking for characteristic bands that indicate the presence of a Functional Group.

This lab manual contains copies of spectra of various compounds. These will serve as the reference spectra for the infrared experiment. Study these spectra and become familiar with the regions that indicate the presence of -OH, C=O, C-H, etc. Where is the "Fingerprint Region" and what is its significance? *Consult your lecture text for help with these questions.*

Make your own Table of Functional Groups and IR Frequency Ranges. (Consult your lecture text for these important numbers)

Some IR stretching frequencies are located here.[2]

[2] ../typicalIR.pdf

PRE-LAB

Answer all assigned WebAssign questions.

Questions

1 What part of the electromagnetic spectrum is known as the infrared region? Express your answer in terms of both frequency (wavenumbers) and wavelength (microns). You may need to consult the lecture text on this one.

2 What is the frequency range of the characteristic absorption bands of the following bonds?

(a) C=O _____

(b) O-H _____

(c) C=C _____

(d) C-C _____

3 Describe two significant differences between the infrared spectra of ethyl alcohol and ethylene.

4 Material safety data sheets (MSDS) for the chemicals used in the lab can be found in:

http://www.ncsu.edu/ncsu/ehs

From my TA

In the lab

5 In case of fire, the recommended evacuation route is:

Down the nearest stairs to the first floor, meet the rest of the class at the front of the building

Take the elevator to the basement and leave on my own

Take the elevator to the first floor, meet the rest of the class at the front of the building

6 Which region in the IR spectrum could be used to distinguish between butanoic acid and 2-butanone?

(a) 3200-3600 cm^{-1}

(b) 1600 cm^{-1}

(c) 1680-1750 cm^{-1}

(d) 2500-3300 cm^{-1}

7 Which is the order of increasing bond stretching frequency (lowest first)?

(I) C-H

(II) N-H

(III) O-H

(IV) F-H

PROCEDURE

1 Your objective with this experiment is to find out the identity of your unknown compound. It can only be one of the following options:

Alcohols:

benzyl alcohol

2-pentanol

Aldehydes/Ketones:

acetophenone

2-chlorobenzaldehyde

4-methyl-2-pentanone

Hydrocarbons:

toluene

n-hexane

1-hexyne

cyclohexene

2 *After* you have carefully listened to the instructions from your instructor and have taken notes on how to operate the instrument, you are ready to take your first IR spectrum. The easiest way to take the spectrum of a liquid is without any added solvent. This is said to be a "neat" sample—a sample with nothing added—just the liquid alone.

3 You can also watch a video[3] that describes how to take an IR spectrum using our instruments.

4 Add about 2 drops of your dry liquid sample to the center of the plate on the IR spectrometer. Make certain that ALL instructions given by your instructor (or the lab video) have been followed.

5 Take your spectrum, print it, and record on it the wavelength of significant peaks (the most prominent peaks in the spectrum).

6 On your spectrum, record the sample name, date, your name, and other relevant information.

7 Rinse the IR Spectrometer plate with dry acetone, dry with a clean Kimwipe, and place used Kimwipes in the unwanted materials container.

8 Try to assign *at least three* of the most prominent bands in the spectrum. Are they consistent with the Functional Group you believe is present in your sample? (Consult the Table of Typical IR Frequencies[4] in the IR section of your textbook.)

[3] https://www.youtube.com/watch?v=4IYMNSRCqy0
[4] ../typicalIR.pdf

IN-LAB QUESTIONS

The laboratory work involves identification of an unknown by recording its infrared spectrum, investigating the major absorption bands, and comparing the spectrum with spectra of a group of known compounds.

Please print the worksheet for this lab. You will need this sheet to record your data.

Questions

1 Record the number of your unknown _____

2 Prepare a spectrum of your unknown and attach the spectrum to this report form.

3 Record the characteristic absorption bands for your unknown.

4 Circle the functional group listed below which is suggested as a candidate for identification by a major absorption band in the spectrum of your unknown.

alcohols
benzyl alcohol
2-pentanol

aldehydes/ketones
acetophenone
2-chlorobenzaldehyde
4-methyl-2-pentanone

hydrocarbons
toluene
n-hexane
1-hexyne
cyclohexene

amines/phenols
aniline
o-cresol
di-n-propylamine

5 Compare the spectrum of your compound with the spectra of the compounds in the functional group that you have circled. Reference spectra may be found in the lecture text or the laboratory manual. Identifying other major peaks may help you isolate the compound. Write the name of the compound you believe your unknown to be.

