INFRARED SPECTROSCOPY

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Introduction

Infrared spectroscopy deals primarily with the internal vibration and/or rotation of molecules as a result of the absorption of infrared light (heat). A group of atoms within a molecule will vibrate at a particular frequency, either in stretching or bending mode (see examples below). All the atoms in a molecule will be involved in at least one group that is stretching and/or bending at any given time. The molecule (actually, it is the group of

stretching bending

atoms) will absorb infrared energy that corresponds to the vibrational frequency exhibited by that group (remember E=hν). Many functional groups show absorption of IR energy at particular wavelengths, with little change from molecule to molecule--this type of absorption is called a *GROUP FREQUENCY*. A group frequency is nearly constant, no matter what other atoms are present in a molecule; if it does change, it does so in a fairly predictable manner.

The appearance of an infrared spectrum is somewhat bewildering at first. First, there are usually two "scales" on each of the axes. Then, the absorption bands (often called peaks) are upside-down. And there are usually many peaks. This is because the spectrum reflects many (but not all) of the vibrational and/or rotational absorptions occurring in the molecule as the spectrometer scanned over a particular wavelength range. Because the interactions between all the different vibrations in a molecule can be quite complex, the spectrum looks complicated. But don't be intimidated by all the absorption bands--not all of them are useful, so you will just concentrate on the ones you need to use, primarily the group frequencies.

In this experiment, you will use an infrared spectrum to determine the identity of a liquid unknown. There are six possible unknowns; each contains different functional groups. By using IR to identify the functional groups present in your compound, you will propose a structure for your unknown.

Prelab assignment

Read the IR spectroscopy section of Chapter 14 prior to lab.

Experimental Procedure

1. For each of the six possible unknowns, predict the expected IR absorption bands.

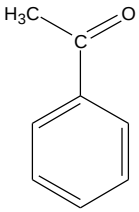
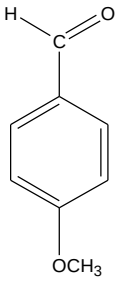
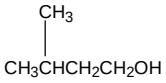
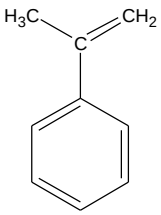
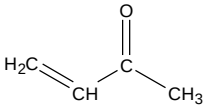
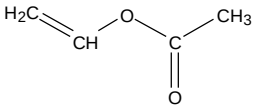
2. Obtain an unknown from the instructor and record its number in your lab notebook. The instructor will demonstrate the correct way to prepare your sample for infrared analysis; pay close attention, as you will probably be preparing your own sample in the future. You will give your sample to the instructor, who will either operate the instrument (FT-IR) and computer for you, or show you how to do it yourself. Clean your salt plates thoroughly with the appropriate solvent (indicated by your instructor), and return them to the dessicator. IT IS IMPORTANT THAT THE SALT PLATES BE KEPT IN THE DESSICATOR WHEN NOT IN USE!!! You will then be instructed how to print your spectrum.

NAME _____ Lab Day/Time _____

INFRARED SPECTROSCOPY LAB REPORT

Unknown Identification Code_____

1. Complete the table below.

Compound	**Expected Absorption Bands**
 acetophenone	
 *p*-anisaldehyde	
 isoamyl alcohol	
 α-methylstyrene	
 methyl vinyl ketone	
 vinyl acetate	

NAME Lab Day/Time

2. What is the structure of your unknown? Is the IR spectrum consistent with your predictions? Explain.

Various stretching frequencies		**(cm^{-1})**
aliphatic C–H		2850 - 2960, 1350 - 1470
aromatic & unsaturated C–H		3000 - 3100
aldehyde C–H		~2750 AND ~2850
≡C–H		3300
triple bonds	C≡C	2100 - 2250
	C≡N	2210 - 2260
double bonds	C=C	1640 - 1680 (m to w)
	aromatic ring C=C	~1500 AND ~1600
	C=O	1690 - 1760 (s)
alcohol & phenol O–H (free)		3610 - 3640
(H-bdd)		3200 - 3600 (s)
carboxylic acid O–H		2500 - 3000 (v br)
amine	R_2NH	3300 - 3400 (ONE peak)
	RNH_2	3300 - 3400 (TWO peaks)
nitro group		1300 - 1390 AND 1500 - 1600 (s)

Double bond out-of-plane bending	
$RCH=CH_2$	910 - 920 AND 990 - 1000
cis-RCH=CHR	675 - 730 (variable)
trans-RCH=CHR	965 - 975
$R_2C=CH_2$	880 - 900

Aromatic rings out-of-plane bending	
monosubstituted ring	690 - 710 AND 730 - 770
ortho disubst'd ring	735 - 770
meta disubst'd ring	690 - 750 AND 750 - 810
para disubst'd ring	810 - 840

C–O stretching frequencies	
primary alcohols	~1050
secondary alcohols	~1100
tertiary alcohols	~1150
phenols	~1230
aliphatic ethers	1060 - 1150
aromatic & vinyl ethers	1200 - 1275 (& 1020 - 1075, weaker)

Carbonyl stretching frequencies	
aldehydes	1720 - 1740
ketones	1705 - 1725
carboxylic acids	1700 - 1725
esters	1730 - 1750
amides	1640 - 1670
acid chlorides	1800
anhydrides	1810 AND 1760

Notes: w = weak; m = medium; s = strong absorptions; br = broad band
conjugation lowers frequency of absorption, attachment to an electronegative substituent increases frequency