

# 10. Infrared Spectroscopy

Organic chemists use infrared spectroscopy (IR) as a means of identifying compounds and determining their structure. It is usually used in combination with other techniques, especially nuclear magnetic resonance spectroscopy (NMR). IR is usually used to determine what types of functional groups are present in the compound and what type of environment the functional groups are in.

In this experiment, you will learn how to use a spectrometer to obtain an IR spectrum and be introduced to the interpretation of IR spectra. You will obtain the spectrum of an unknown compound. You may also identify unknown compounds from spectra distributed by your instructor.

## **PRE-EXPERIMENT ASSIGNMENT**

Study this chapter of the manual, the lecture notes on the Chemistry Department web site, pages 194-215 in Williamson, and the appropriate section in Solomons and Fryhle, your lecture textbook. Consult the index to find the IR section in the edition you are using. Do the first parts of your notebook writeup.

**A student who has prepared for the Infrared Spectroscopy experiment should be able to:**

1. Identify the relative energy, frequency, and wavelength of infrared light compared with other types (including gamma rays, x rays, ultraviolet and visible light, microwaves and radio waves). Also, identify the change that occurs in molecules when infrared light is absorbed.
2. Define, identify, recognize, and explain the use of each of the following: IR (infrared) spectroscopy, neat samples, salt plates (including how to clean them), Nujol mull, KBr pellet
3. Draw the structure given the name, or give the name from the structure, of the compounds used in the day's experiment, and give the role of each (reactant, solvent, catalyst, etc.).
4. Identify and explain safety considerations for this experiment.
5. Perform the day's experiment safely and successfully.

Quizzes given after the experiment has been performed may also include:

6. Give or recognize a probable absorption frequency in  $\text{cm}^{-1}$  in the IR spectrum of a compound containing any of the following functional groups. (You may also be given a chart to help you identify other functional groups. A copy of this longer chart is included at the end of this chapter.)

Functional Group	Approximate Frequency ( $\text{cm}^{-1}$ )	Peak Description
O-H (hydrogen bonded alcohols and phenols)	3200-3550	strong, broad
O-H (hydrogen bonded carboxylic acids)	2500-3000 or above	variable, very broad
N-H	3300-3500	medium
C=O	1630-1820 (most 1700-1780)	strong

7. Determine the presence (or absence) of functional groups in a sample from infrared spectral data. The data may be given in the form of a list of peak locations (see #6 above) or as a copy of an actual spectrum.

### Obtaining the Spectrum

You will obtain the spectrum of an unknown compound. Your unknown will be one of these four substances: propanoic acid, 2-propanol, cyclohexanone, and cinnamaldehyde. Because the method depends on the particular instrument you use, we will not describe the procedure here. You will be shown how to prepare your sample and operate the instrument in the laboratory.

If you obtained an IR spectrum during a previous laboratory, bring it with you to lab this week. Your instructor will give a lecture on infrared spectroscopy. If you have not yet obtained a spectrum, you will do so after your instructor's lecture.

As soon as you have recorded your spectrum, write your name, the name of the compound or the number of the unknown, and the date on any spectrum (this is *annotating* the spectrum).

### POST-EXPERIMENT ASSIGNMENT

Write the lab report and have it ready to turn in by the beginning of the next lab. Include your spectrum with the lab report; it is your data.

To identify your unknown, first make a table of the peaks in your IR spectrum. Most important are those at or above  $1600 \text{ cm}^{-1}$ . Give the

location of each peak as a single number, not a range. ***The positions you indicate for peaks must be those in your spectrum, not the general regions given in the tables in this manual, Williamson, Solomons, or any other table.***

Look up the structures of the four possible compounds the unknown might be, if you do not know them already, and identify the functional groups present in these structures. Match the functional groups that give peaks in the IR spectrum of your unknown with the functional groups present in one of the four compounds. This is how you identify your unknown. There will be many peaks on your spectrum that you will not assign to any functional group. This is normal. Include the name and structure of your unknown in the Conclusions section of your final report, and explain how you arrived at this identification.

Prepare for the infrared spectroscopy portion of the next quiz.

### Simplified Table of Main IR Frequencies

Wave number, $\text{cm}^{-1}$	Functional Group	Peak Description
3300 – 3600	O-H (alcohol)	Strong and broad
2500 – 3000 can reach	O-H (carboxylic acids)	Very broad (over $\sim 500 \text{ cm}^{-1}$ ), often looks like distorted baseline, above $3000 \text{ cm}^{-1}$ .
3200 – 3500	N-H	Doublet in case of $\text{NH}_2$ group of a primary amine or amide
3300	$\equiv \text{C} - \text{H}$ terminal alkyne	Usually sharp and strong
3000 - 3100	$= \text{C} - \text{H}$ alkene or arene	Often weak, overlaps with CH alkane absorption
2800 – 3000	C-H ( $\text{sp}^3$ carbon)	Strong, broad and multi-banded
2250 - 2220	$\text{C} \equiv \text{N}$	Medium intensity
2100 - 2260	$\text{C} \equiv \text{C}$ alkyne	Medium intensity for terminal alkynes, very weak for internal
1680 – 1820	$\text{C}=\text{O}$ (amides, ketones, aldehydes carboxylic acid, esters)	Very strong; lower frequency for amides and when $\text{C}=\text{O}$ is conjugated
1600 – 1650 it's	$\text{C}=\text{C}$ alkene, aromatic ring	Check to see if you have C-H unsaturated $>3000 \text{ cm}^{-1}$ (if not, completely substituted)
$\sim 1600$ corresponding N-H peak otherwise)	$-\text{NH}_2$ (bending) $1^\circ$ amines and amides	Only if you have peak at $3200\text{-}3500 \text{ cm}^{-1}$ (this may be mistaken for $\text{C}=\text{C}$ )
1200 $\text{C}=\text{C}$ first)	Ar-O	Strong (look for $=\text{C}-\text{H}$ &
1050-1150	C-O	
690 and 750	phenyl group	Strong (look for $=\text{C}-\text{H}$ & $\text{C}=\text{C}$ first)