

Fourier Transform Infrared Spectroscopy (FTIR)

Main objectives:

- Understand the fundamental of Infrared Spectroscopy
- Identify unknown sample from their spectra

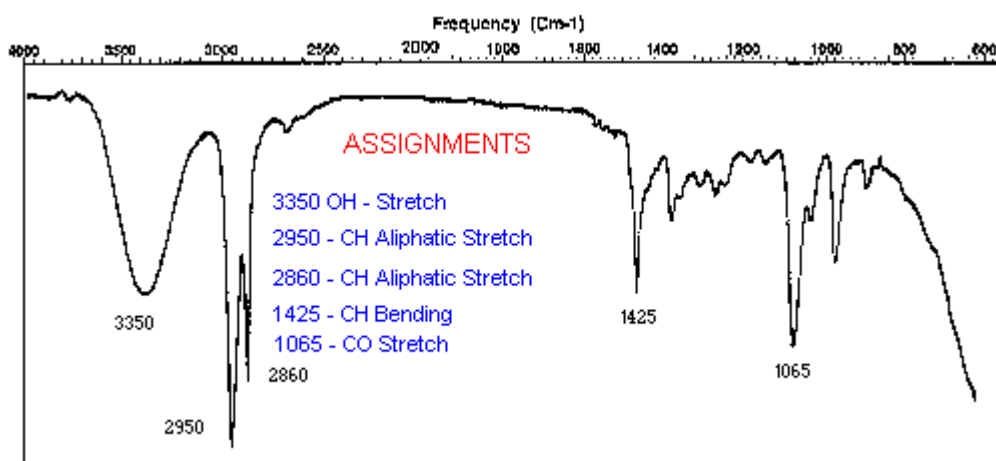
Overview of IR spectroscopy:

IR spectroscopy is an important relatively inexpensive and efficient analytical method for characterizing materials. In this lab we will focus on the chemical structures of certain substances, however IR spectroscopy is used for obtaining an abundance of information such as thermodynamic data, bond length and diffusion data.

On the other hand IR spectroscopy is limited in the ability to obtain accurate quantitative measurements and the range of inorganic materials that are IR visible.

The spectrum that is obtained from IR spectroscopy is due to the vibrational modes of the molecules. Each molecule has its own distinct quantized vibrational energy level. This is often termed its 'fingerprint'. In FTIR, electromagnetic radiation in the infrared region is directed at the sample. When the frequency of this energy matches the frequency of the vibration of the molecules, radiation is absorbed (or transmitted).

Below is an example of an IR spectrum.



Ref.[1]

Some equations:

The relationship between frequency and wavelength is

$$\nu = \frac{c}{\lambda}$$

Where ν is the frequency, c is the speed of light and λ is the wavelength

For wavenumber $\tilde{\nu}$

$$\tilde{\nu} = \frac{\nu}{c} \quad \text{and} \quad \tilde{\nu} = \frac{1}{\lambda[\text{cm}]}$$

The wavenumber scale is directly proportional to the energy and vibrational frequency of the molecule

$$\Delta E_{\text{vib}} = hc\tilde{\nu} \quad [\text{cm}^{-1}]$$

Where ΔE_{vib} is the vibrational energy level and h is Planck's constant.

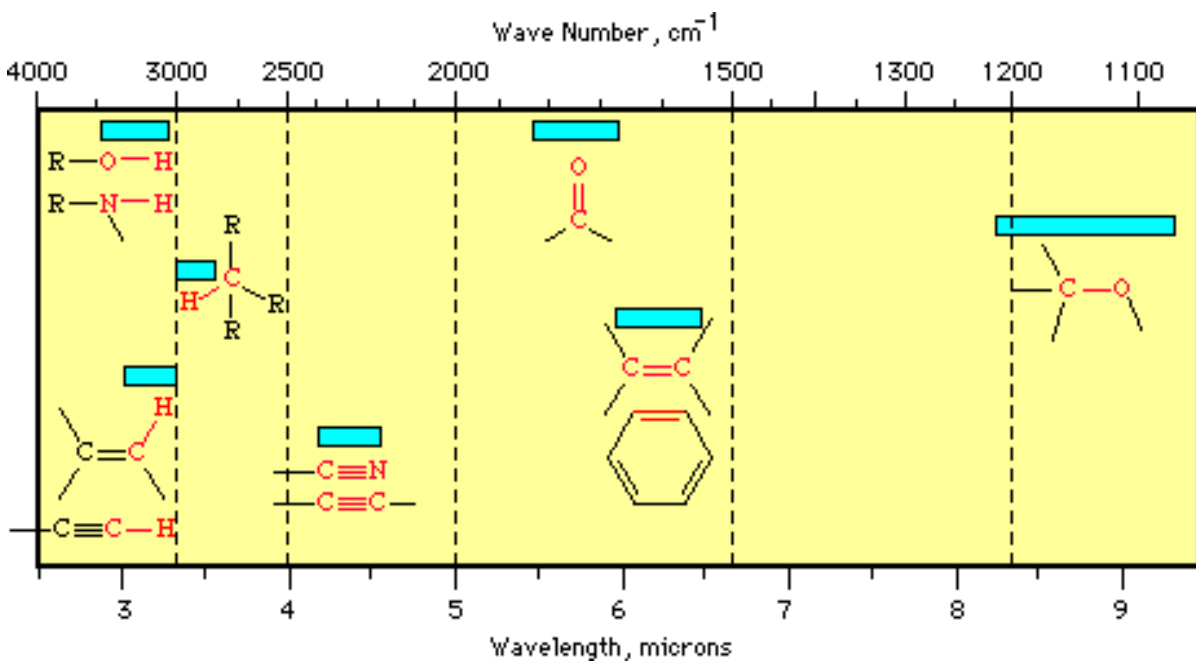
It is important to know that not all materials are IR visible. These are the requirements that must be met called the infrared selection rules:

1. The frequency of the source must correspond to the frequency of the vibration of the molecule.
2. The electric field of the light must interact with the molecules dipole, i.e. there must be an asymmetric net dipole moment.

The change in dipole moment is effected by rotation and vibration of the molecule. This causes the transition of quantized energy states.

Examining FTIR spectra:

Each molecule's fingerprint corresponds to a specific wavenumber. However, shifts in wavenumber can occur. This is due to the interactions with neighboring molecules, i.e. dipole-dipole interactions. Also, isotopes will cause band shifts. On a side note, Nuclear Magnetic Resonance (NMR) will be more sensitive to the presence of isotopes. Below are some examples where the molecules and their respective bondings are located on the IR spectra



Ref [2].

Some information about polymers:

Polymers are compounds consisting of extremely long chains of atoms. As the name implies, they are made up of many identical smaller units called monomers. Monomers are molecules of low molecular weight and usually contain a double bond. Plastics are synthetic polymers built from monomer units as pictured below.

Figure 1: Monomer Units Combine to Form a Polymer

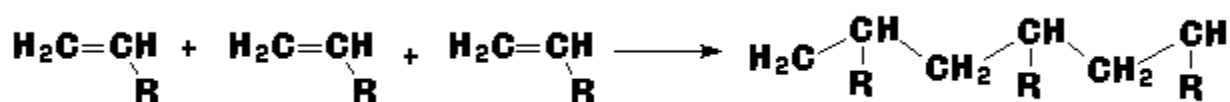


Figure 2: Shorthand Notion of a Polymer



R represents some organic chemical group. For example, R can be a hydrogen, H, a methyl group, CH₃, or a phenyl group, C₆H₅. In the simplest case, the R group is hydrogen and represents the formation of polyethylene. Different R groups attached to the carbon hydrogen "backbone" of the polymer produce different types of plastics.

Table 1: Common Types of Polymers Used in Plastic Films

Type of Polymer	R group (chemical group attached to the C-H backbone)
Polyethylene	-H
Polypropylene	-CH ₃
Poly(acrylonitrile)	-CN
Poly(vinyl acetate)	-CH ₂ COOH
Poly(amide)	-CH ₂ CONH ₂
Polystyrene	-C ₆ H ₅

Ref. [3]

In this lab, you will be asked to draw Lewis structures. Following are some instructions for drawing the Lewis dot structures:

1. Count the valence electrons
2. Draw a skeleton structure for the species, joining atoms by single bonds. The central atom is usually the one written first in the formula. Terminal atoms are most often hydrogen, oxygen or a halogen.
3. Determine the number of valence electrons still available for distribution.
4. Determine the number of valence electrons required to fill out an octet for each atom in the skeleton.

Ref. [4]

Questions to consider:

In wavelength and wavenumber, where does the IR region lie in the electromagnetic spectrum?

A background spectrum is necessary before any samples can be run, what do you expect to see?

Will polar bonds yield weak or strong IR absorption?

Lab assignment:

Two unknown samples will be run during the lab. You will have printouts of both spectra. The following is what you will be required to do for this lab.

1. Identify the peaks by their wavenumber and relative intensities.
2. Correlate the wavenumber to possible bonding (i.e. types of vibrations). There are many IR and polymer characterization books that contain tables with this information. References 8 & 9 have the vibration tables, however, there are many other places to find this data.
3. You will be required to draw the Lewis dot structures of the two unknowns that are run in your lab. It is suggested that once you have found the possible

structures, draw the Lewis dot structures of the groups, this will make the next step easier. Include this in your lab report.

4. Once you have chosen some possible structures that match the wavenumbers, find the polymers that have those structures. Hint: the unknowns will only contain C and H, this will minimize your search quite a bit. There are two reference books, ref. 10 & 11, which you can use that contain lists of polymers and their structures. Again, any other source is fine.
5. Once you have an idea of the possible polymers your unknown could be, try to match the spectra to the reference spectra (use either the Monomers and Polymers IR Grating Spectra reference books by Sadtler or The Aldrich Library of Infrared Spectra by Pouchert). Keep in mind the spectra will not be a perfect match, but by looking at the peak wavenumber, relative shape and intensity, it should be very close. Obtain copies of the spectra of the identified unknowns and attach them to your report.
6. For the write-up, you will follow the same format that was mentioned in class. The most important aspect of the report is your line of reasoning in trying to identify the unknown.

References:

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