



INSTRUMENTAL ANALYTICAL METHODS



INFRARED SPECTROSCOPY

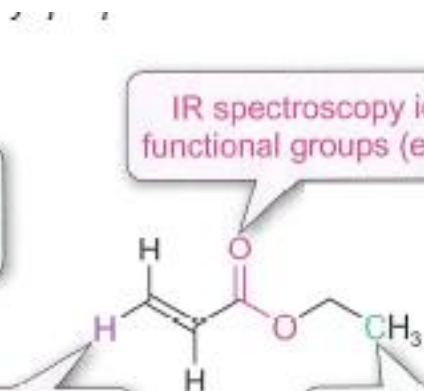


MS determines the relative molecular mass and atomic composition (e.g. $C_5H_8O_2$)

IR spectroscopy identifies functional groups (e.g. esters)

1H NMR spectroscopy gives detailed information on the different types of hydrogen atoms

^{13}C NMR spectroscopy provides information on the carbon skeleton

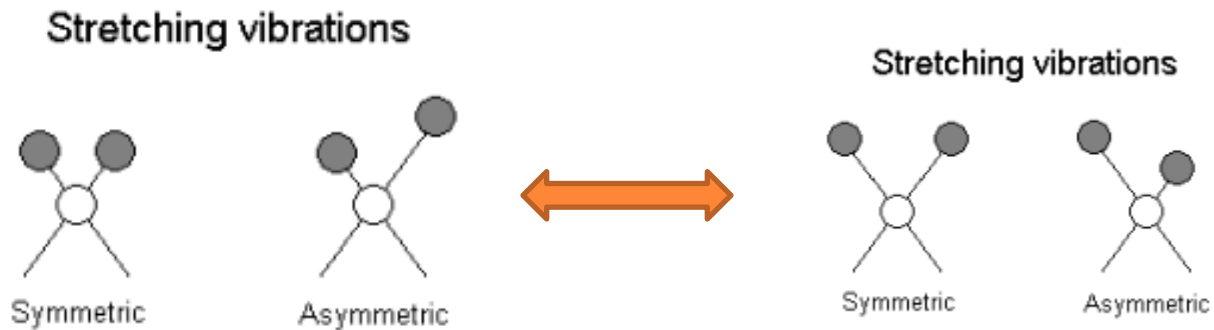


MOLECULAR VIBRATIONS

- The positions of atoms in a molecules are not fixed; they are subject to a number of different vibrations. Vibrations fall into the two main categories of
 - *stretching* and
 - *bending*.

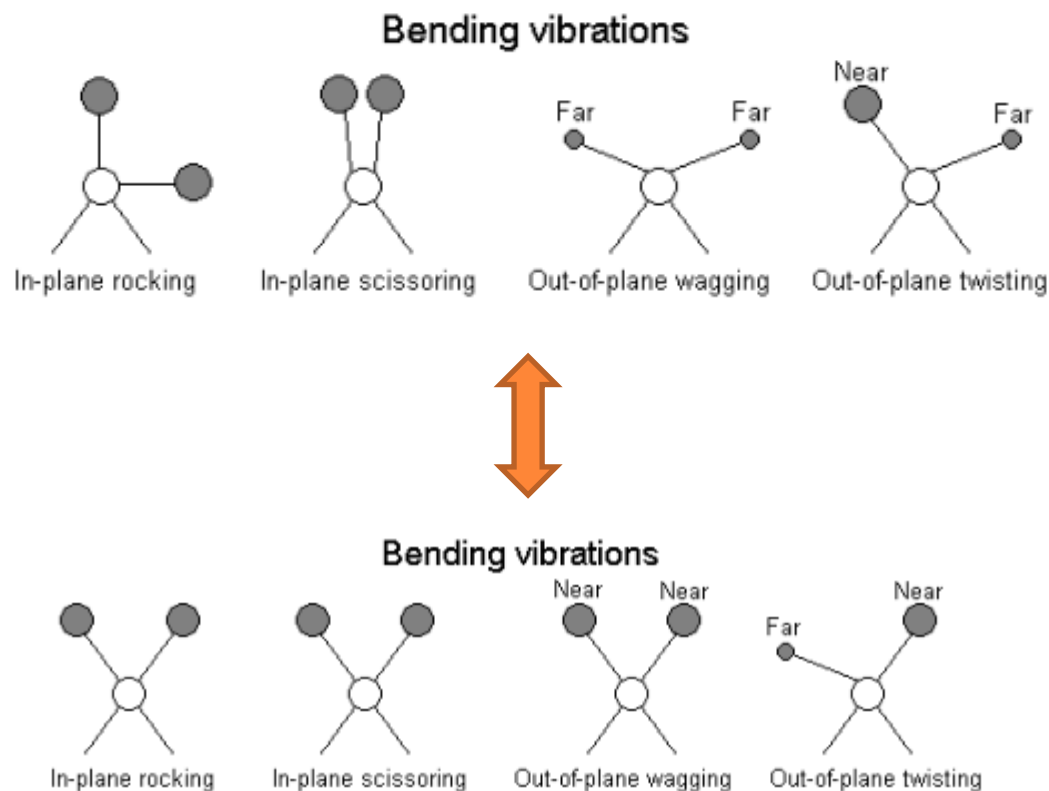


- **Stretching:** Change in inter-atomic distance along bond axis



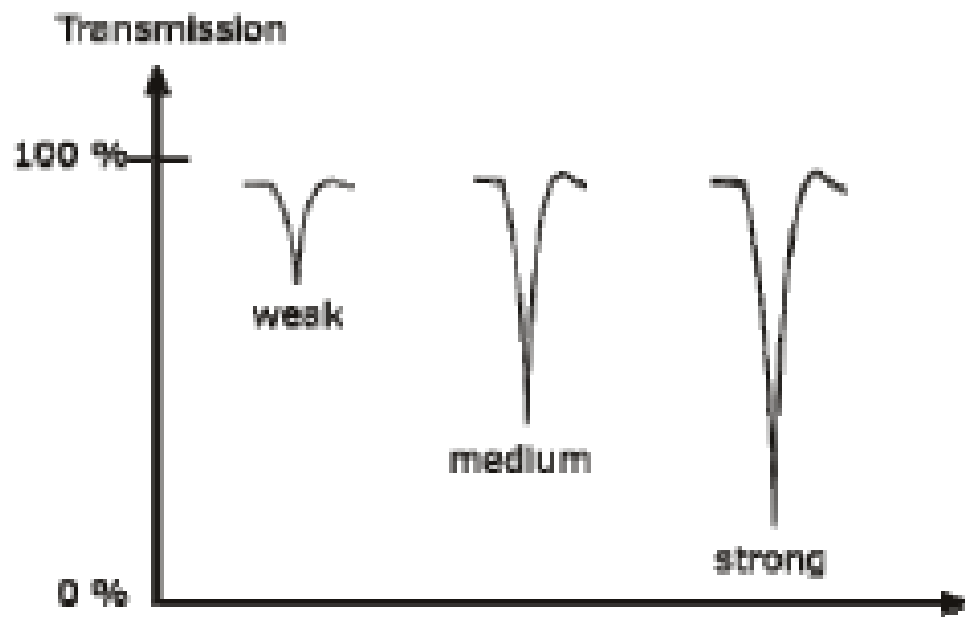
Bending: Change in angle between two bonds.
There are four types of bend:

- Scissoring
- Rocking
- Wagging
- Twisting



CLASSIFICATION OF IR BANDS

IR bands can be classified as strong (s), medium (m), or weak (w), depending on their relative intensities in the infrared spectrum. A strong band covers most of the y-axis. A medium band falls to about half of the y-axis, and a weak band falls to about one third or less of the y-axis.

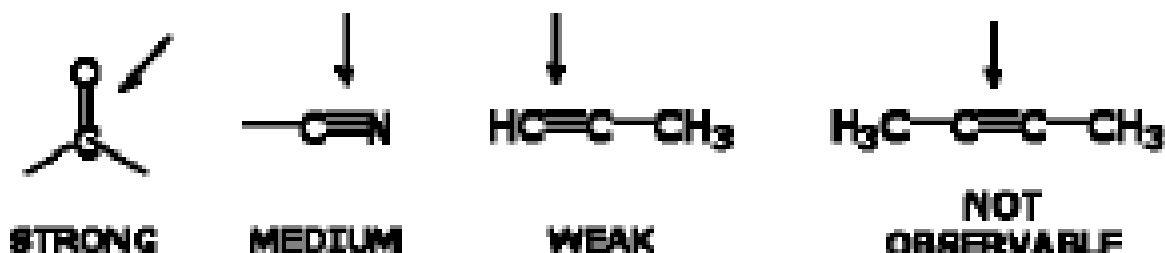


INFRARED ACTIVE BONDS

Not all covalent bonds display bands in the IR spectrum. Only polar bonds do so. These are referred to as IR active.

The intensity of the bands depends on the magnitude of the dipole moment associated with the bond in question:

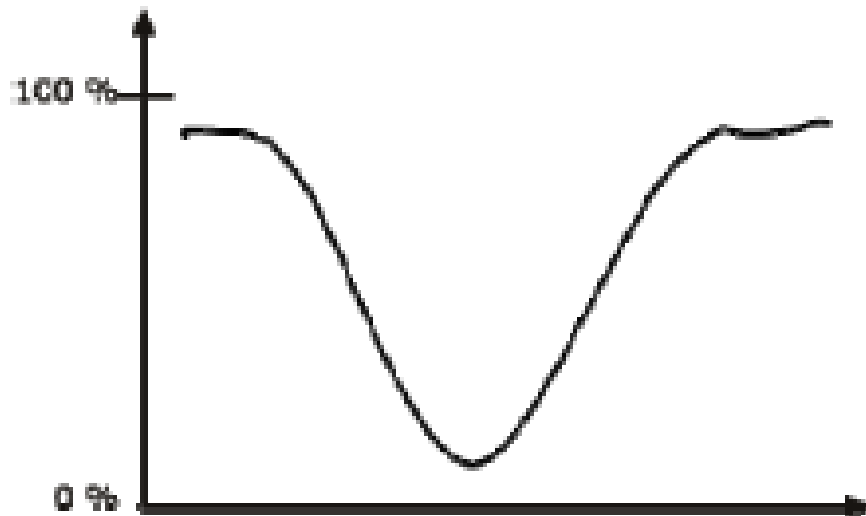
- Strongly polar bonds such as carbonyl groups (C=O) produce strong bands.
- Medium polarity bonds and asymmetric bonds produce medium bands.
- Weakly polar bond and symmetric bonds produce weak or non observable bands.



INFRARED BAND SHAPES

Infrared band shapes come in various forms. Two of the most common are narrow and broad. Narrow bands are thin and pointed, like a dagger. Broad bands are wide and smoother.

A typical example of a broad band is that displayed by O-H bonds, such as those found in alcohols and carboxylic acids, as shown below.



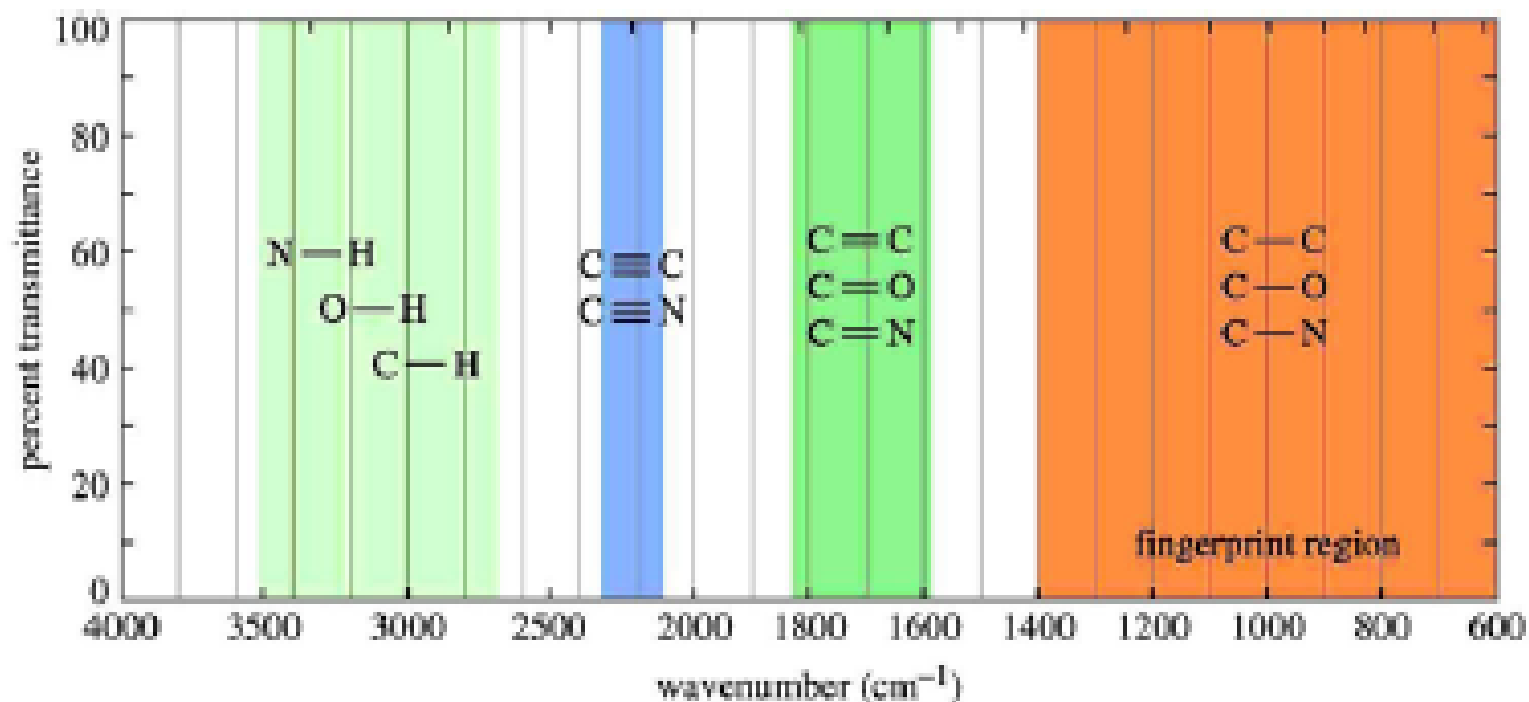
INFORMATION OBTAINED FROM IR SPECTRA

- IR is most useful in providing information about the presence or absence of specific functional groups.
- IR can provide a molecular fingerprint that can be used when comparing samples. If two pure samples display the same IR spectrum it can be argued that they are the same compound.
- IR does not provide detailed information or proof of molecular formula or structure. It provides information on molecular fragments, specifically functional groups.
- Therefore it is very limited in scope, and must be used in conjunction with other techniques to provide a more complete picture of the molecular structure.

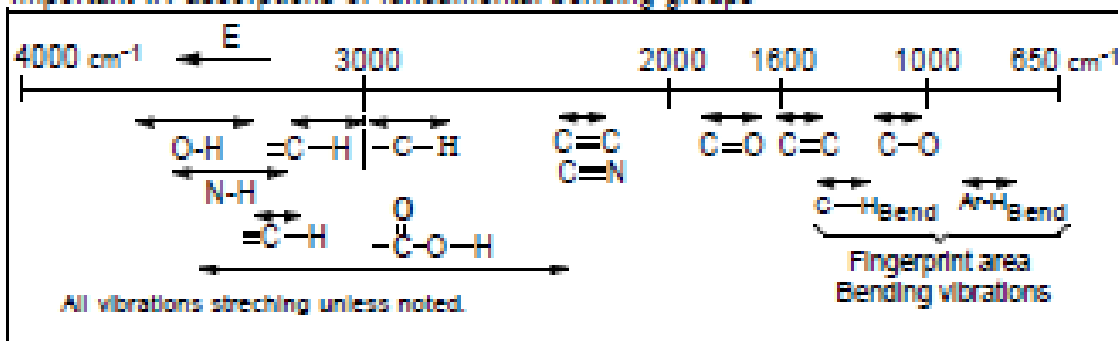


IR ABSORPTION RANGE

The typical IR absorption range for covalent bonds is 600 - 4000 cm^{-1} . The graph shows the regions of the spectrum where the following types of bonds normally absorb. For example a sharp band around 2200-2400 cm^{-1} would indicate the possible presence of a C-N or a C-C triple bond.



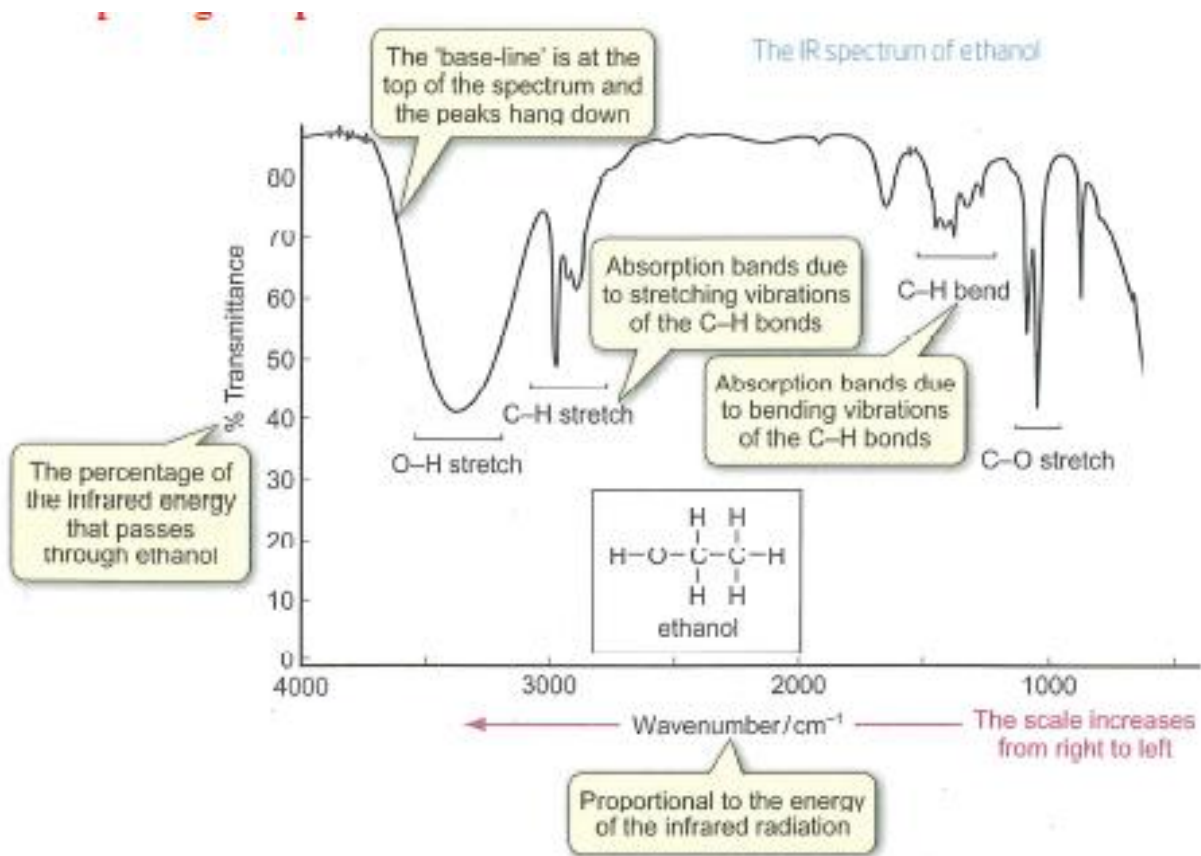
Important IR absorptions of fundamental bonding groups



- Stretching vibrations: 1000 - 4000 cm^{-1} Bending vibrations: 600 - 1600 cm^{-1}
- The fingerprint area gives specified pattern of many peaks (bending vibrations) in the spectra for each organic molecule
- Strong dipoles gives generally strong absorbance, *e.g. Carbonyl, C=O*
- Dipole moment must be changed if IR-radiation should be absorbed.



INTERPRETING IR SPECTRA

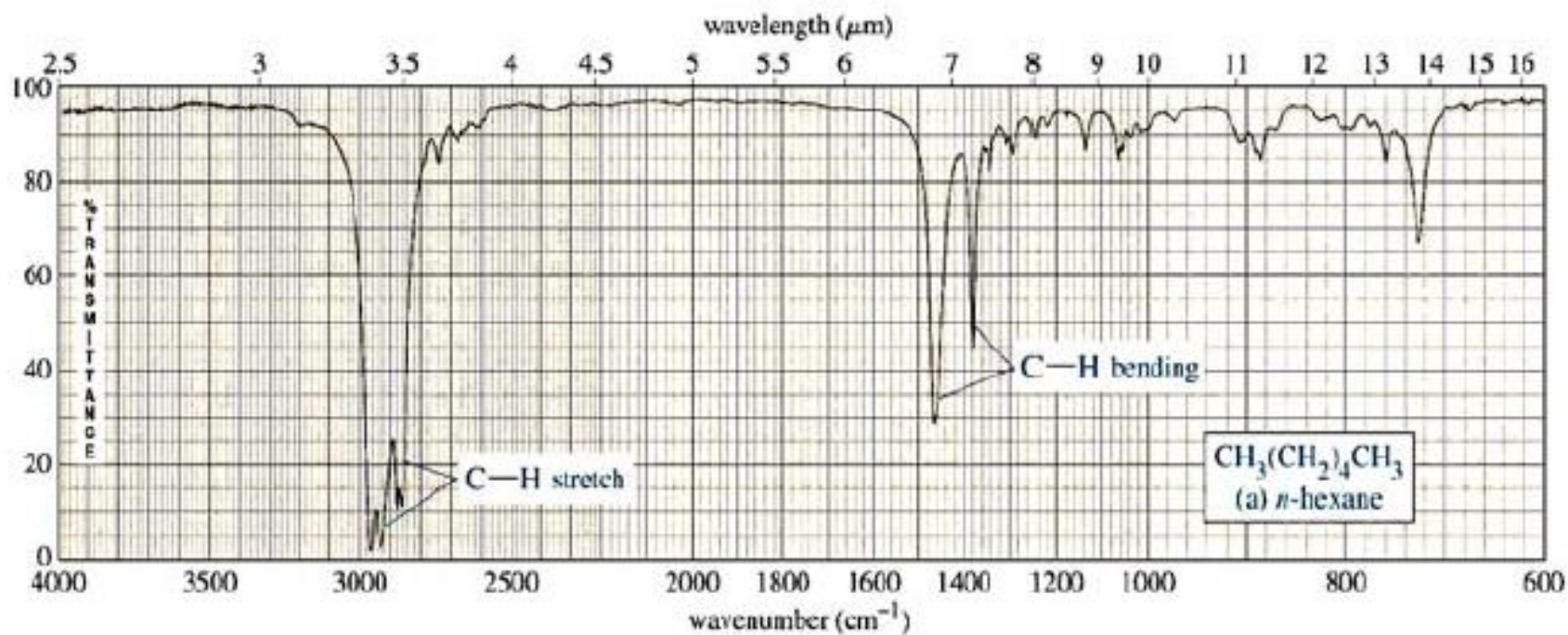


- Generally only certain peaks are interpreted in the IR
- -Those peaks that are large and above 1400 cm⁻¹ are most valuable

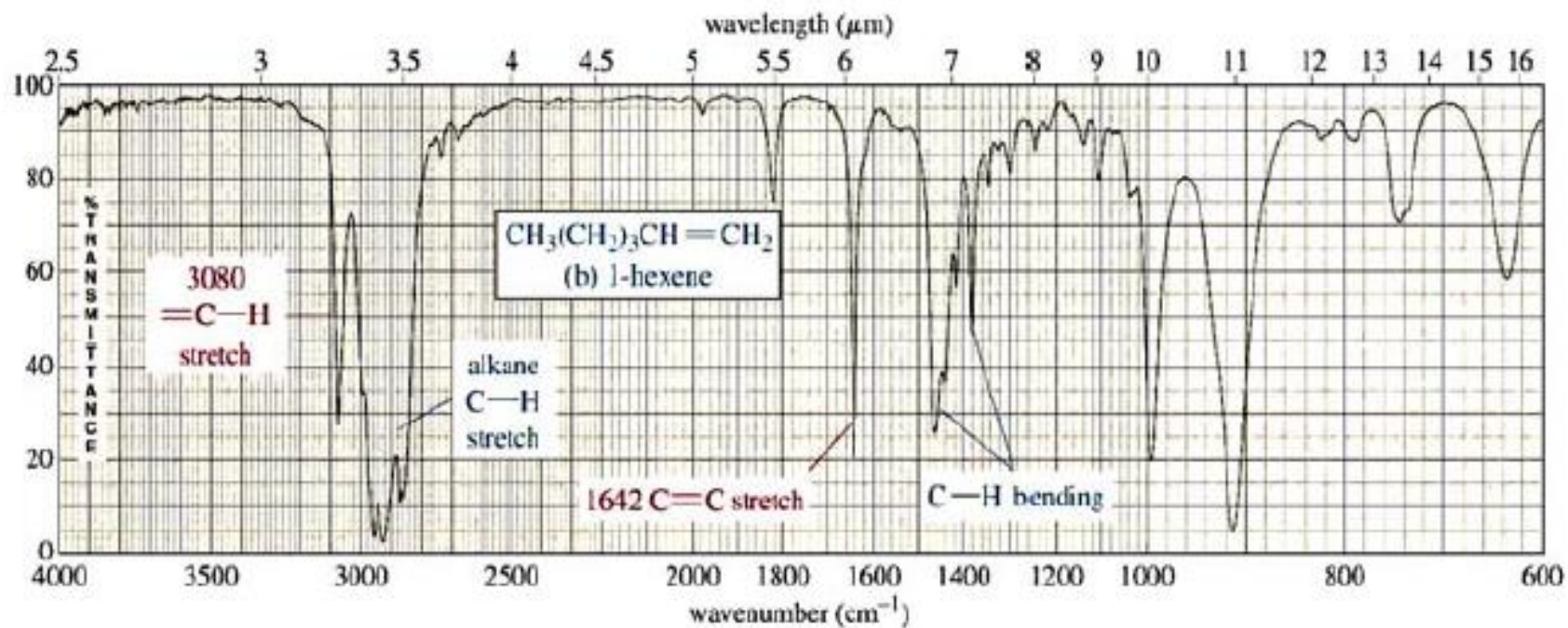


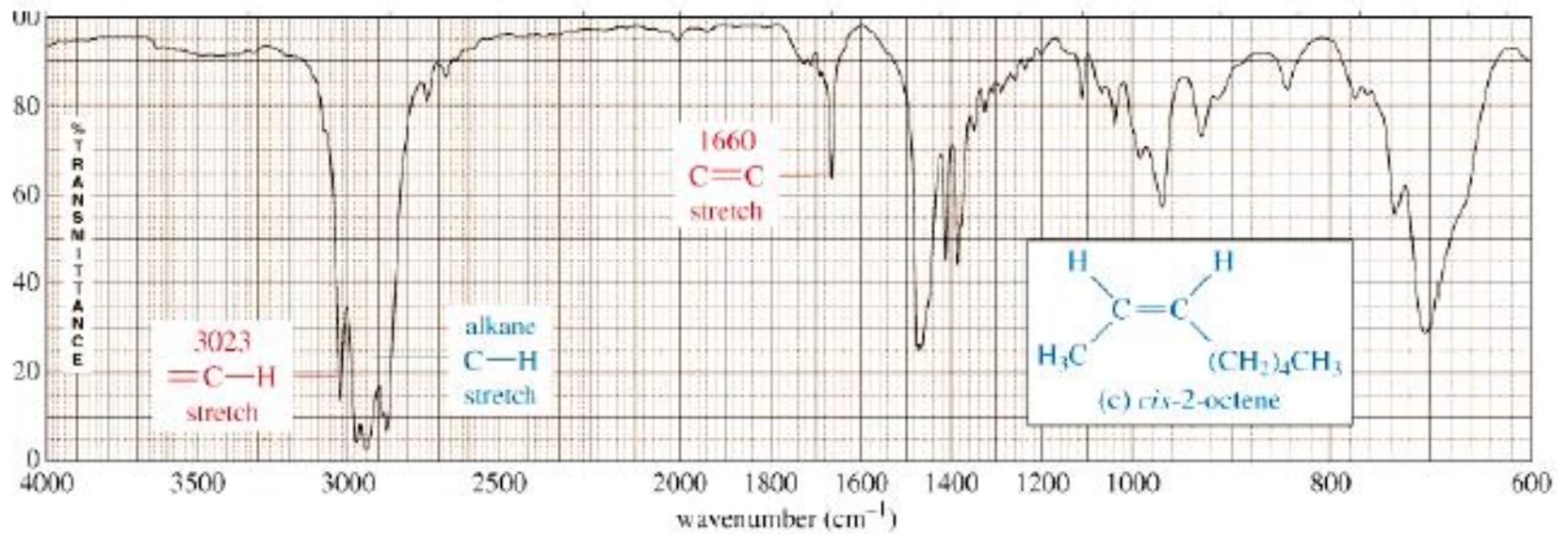
INTERPRETING IR SPECTRA

HYDROCARBONS

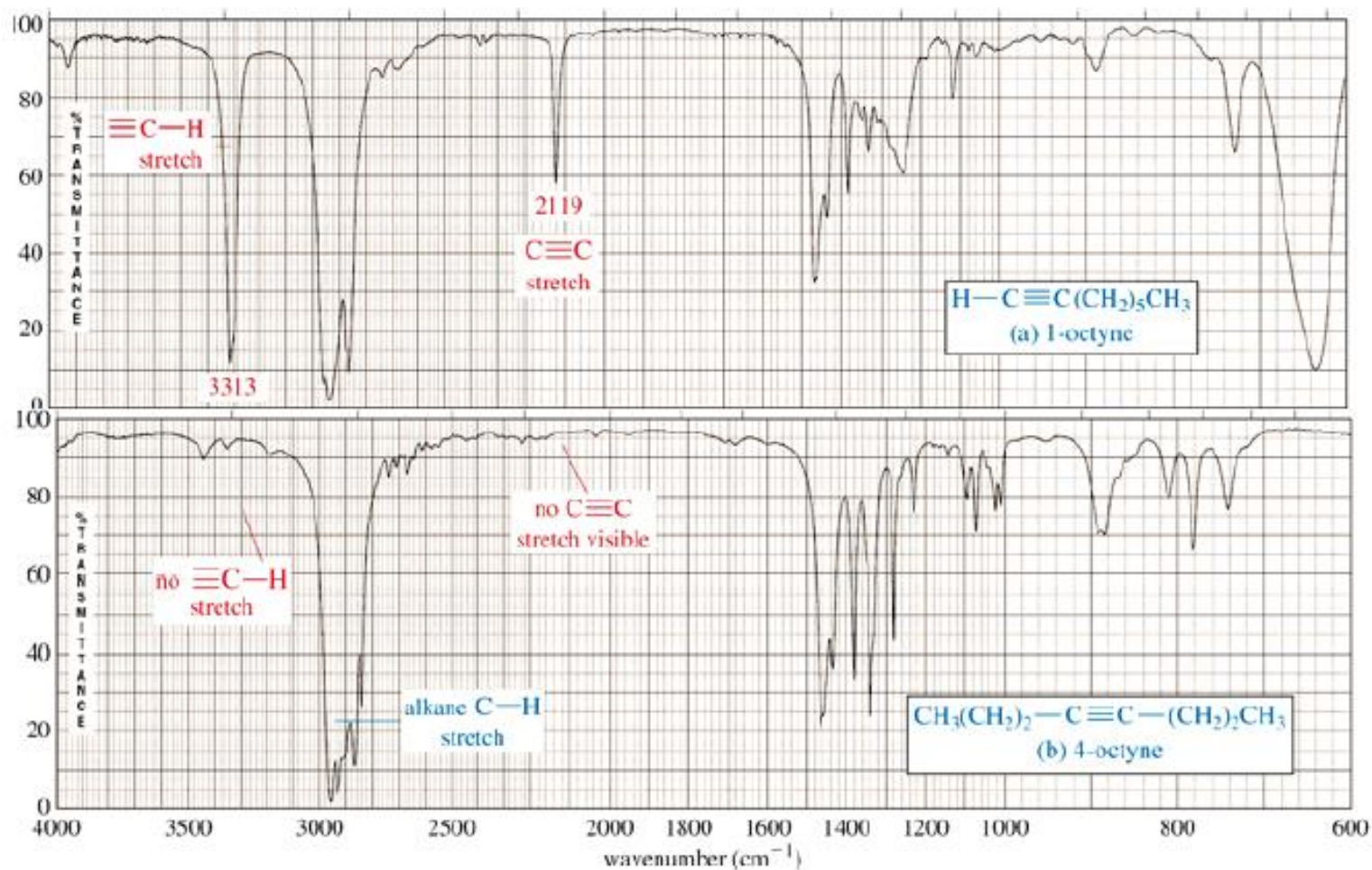


ALKENES

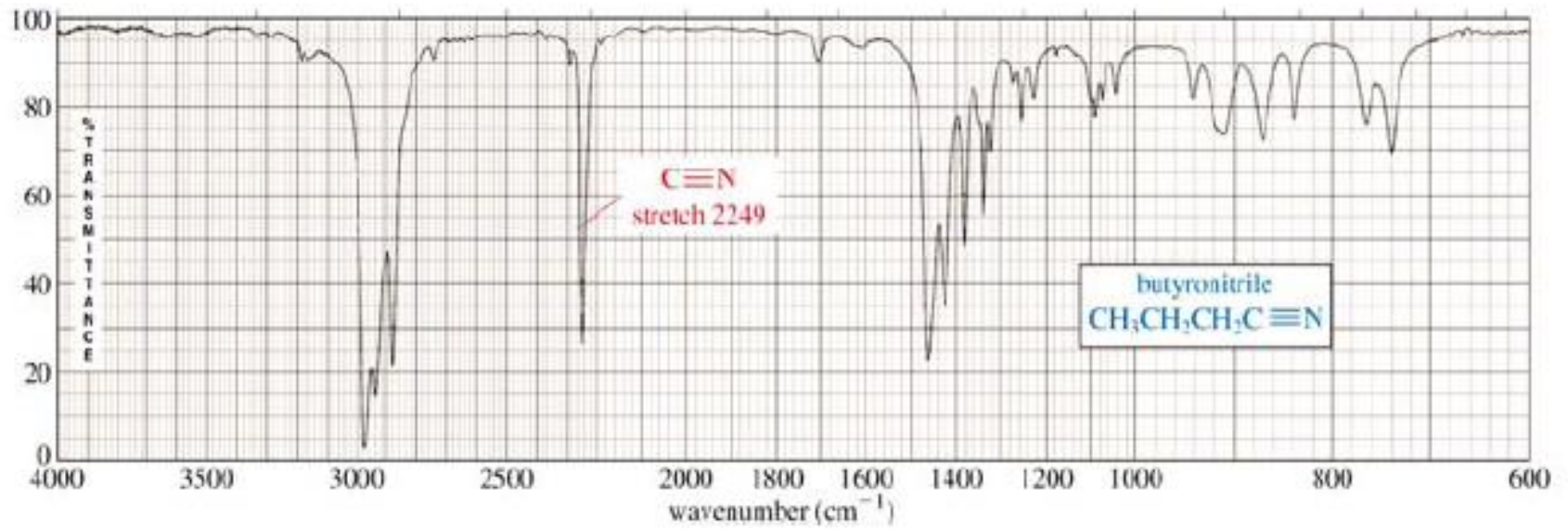




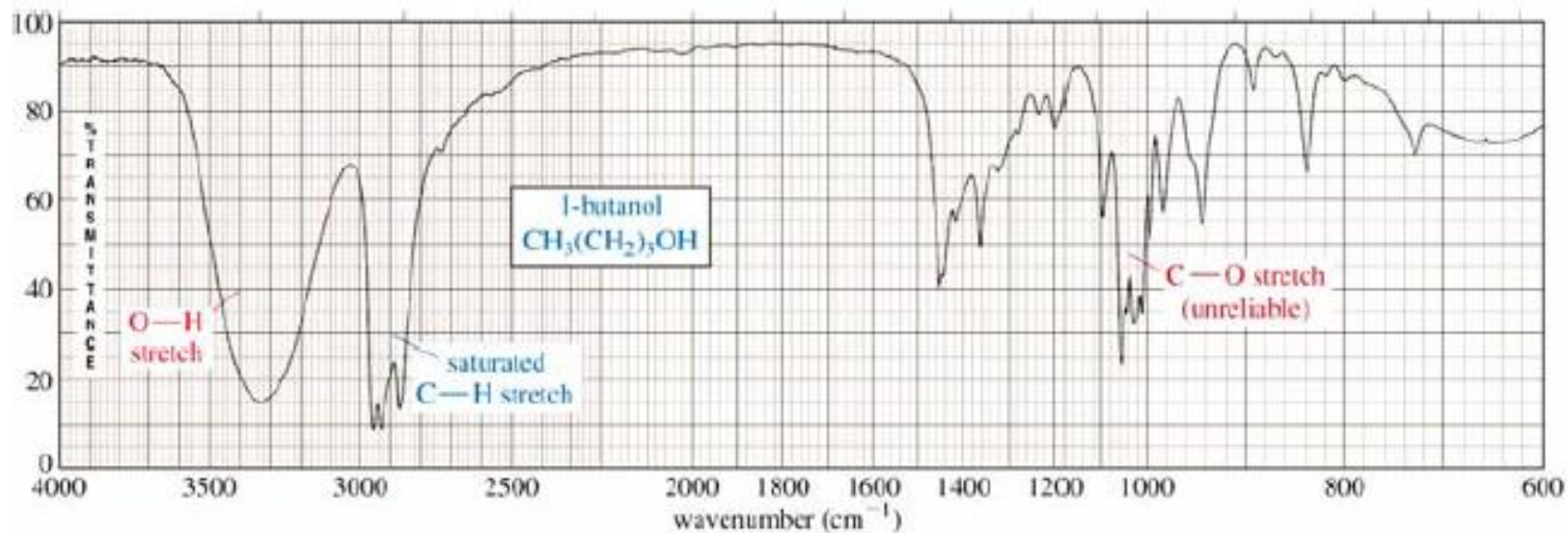
ALKYNS



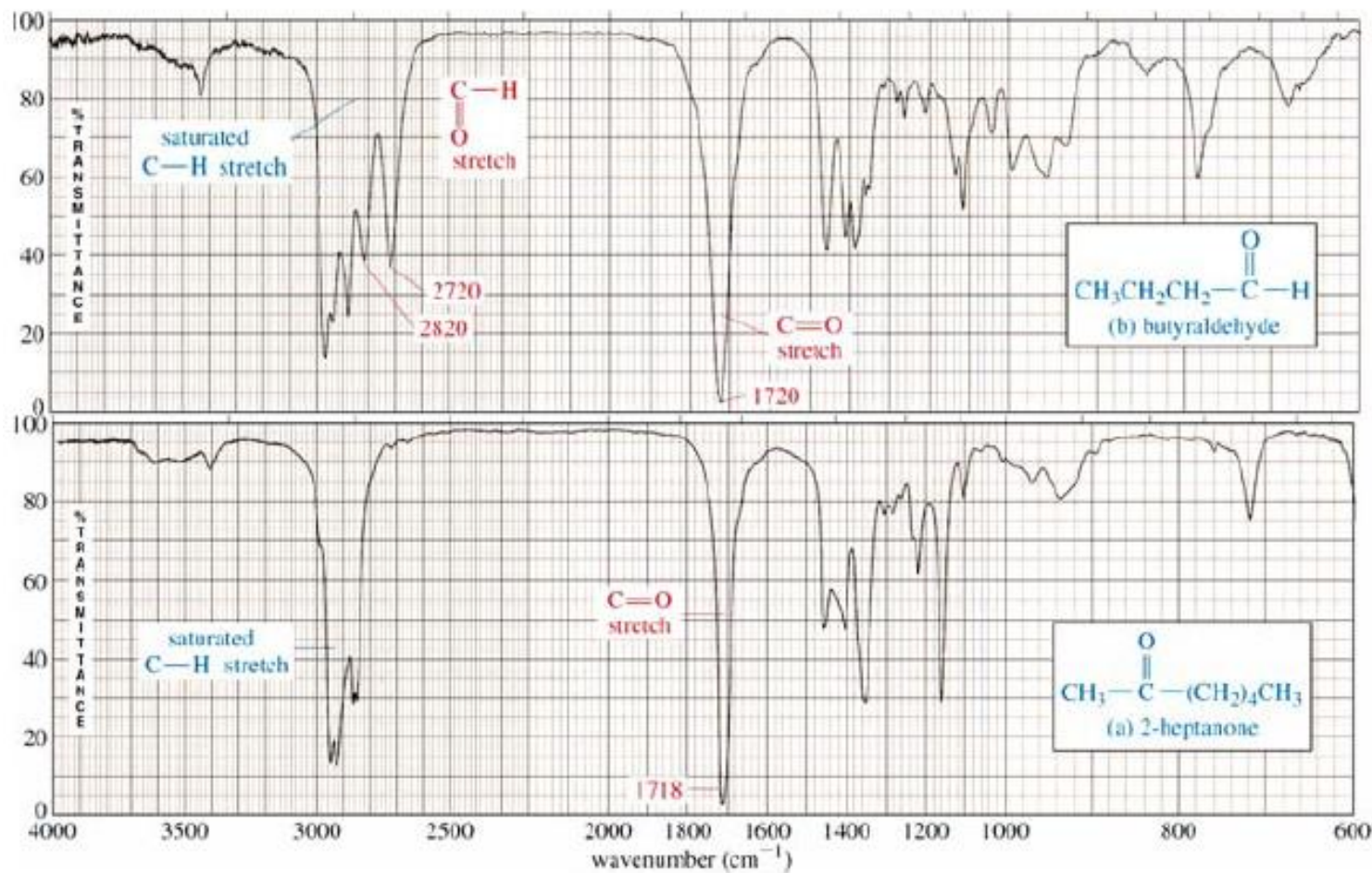
NITRILE



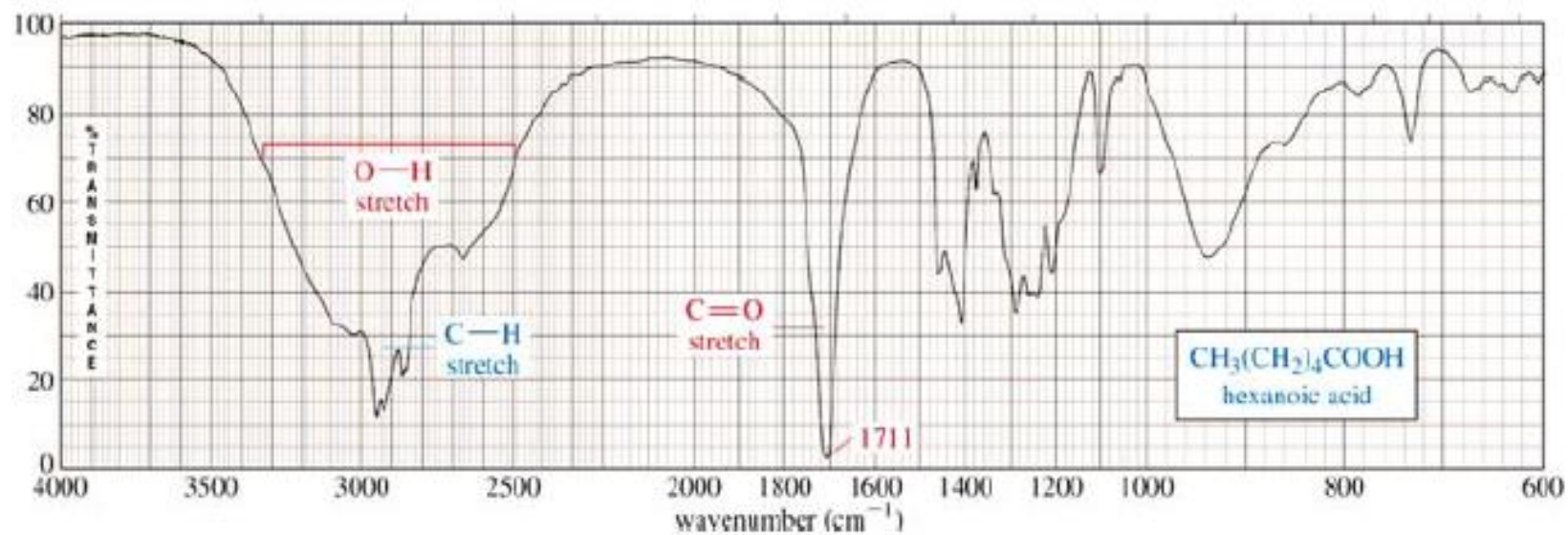
ALCOHOL



IR SPECTRUM OF ALDEHYDES AND KETONES



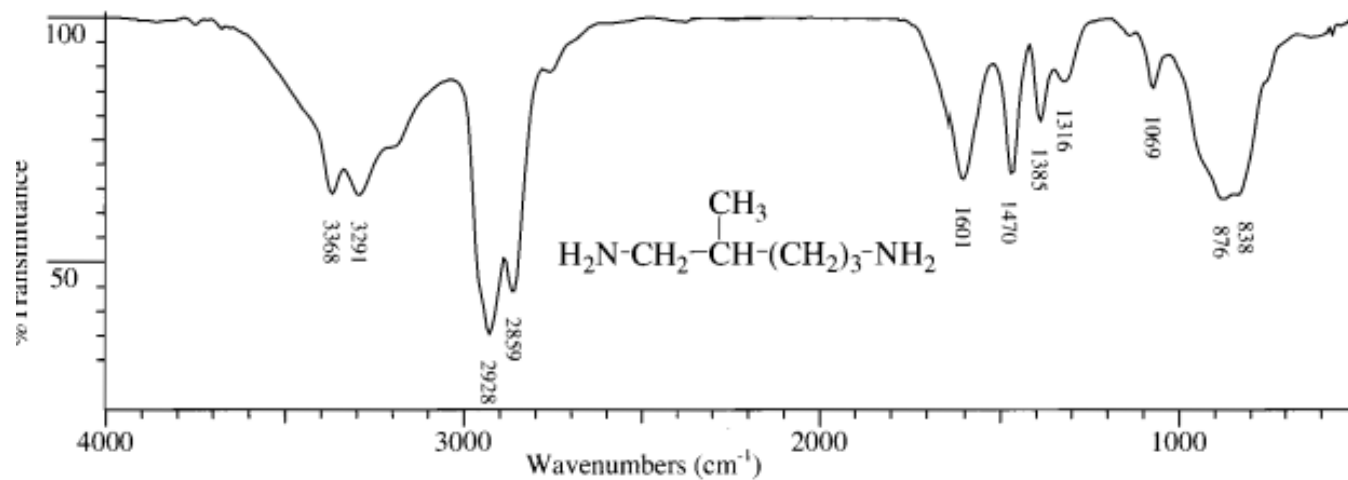
CARBOXYLIC ACIDS



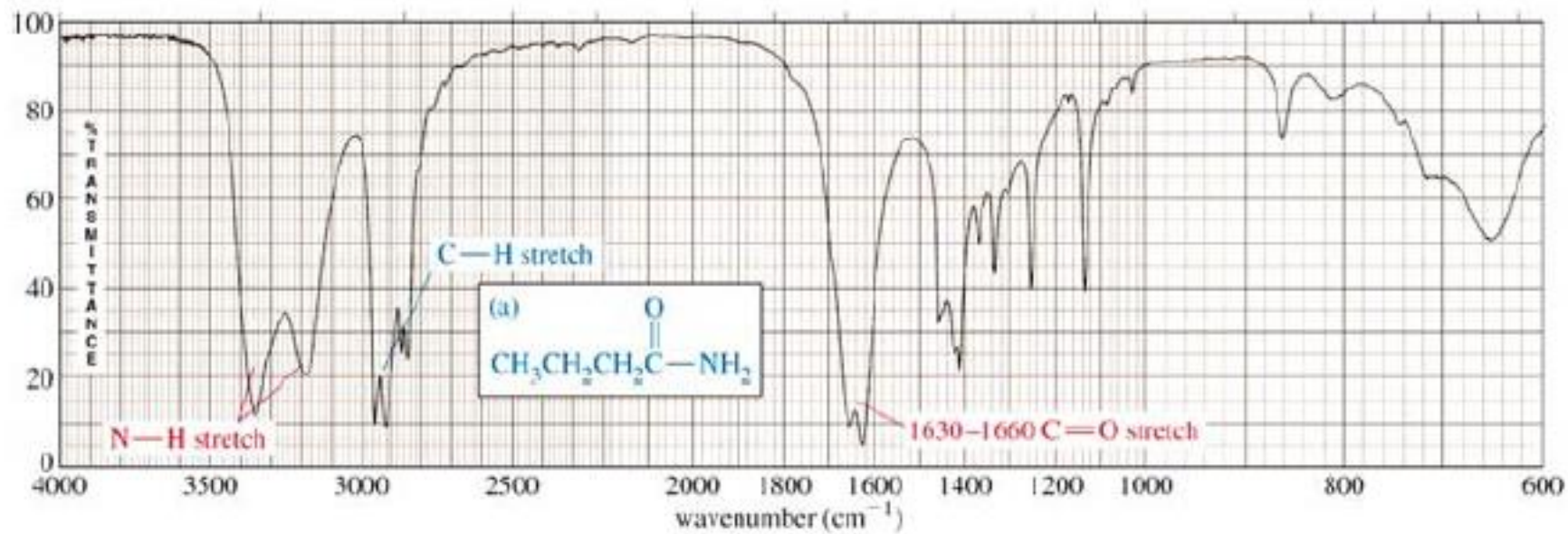
AMINES

Check for N-H.

- Medium absorption(s) near 3400 cm^{-1} .

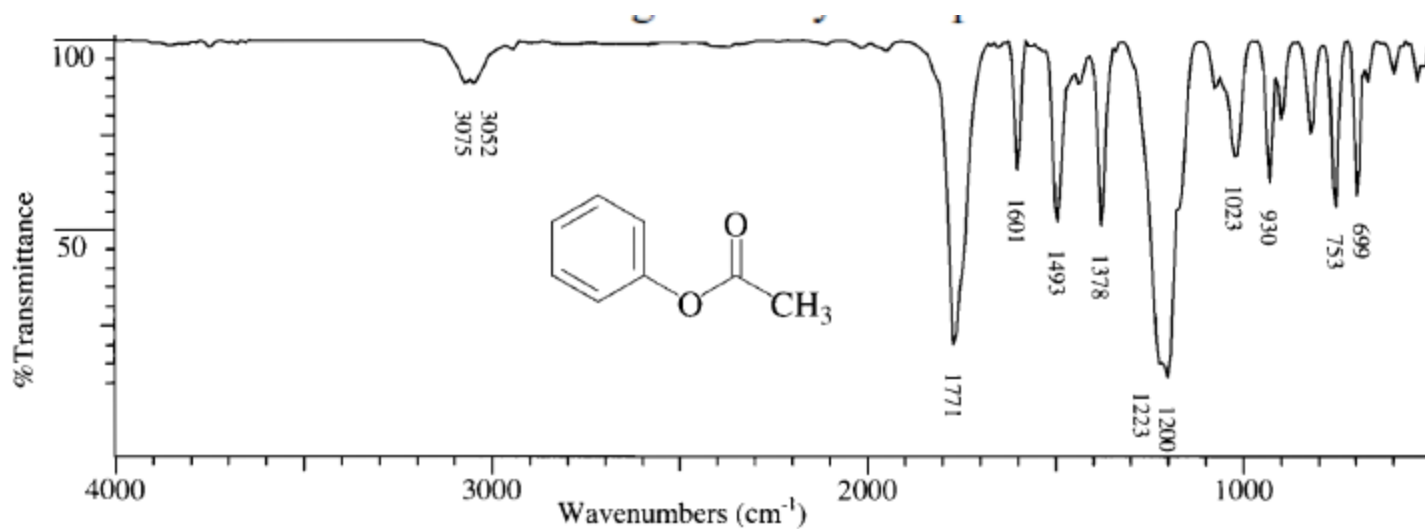


AMIDES



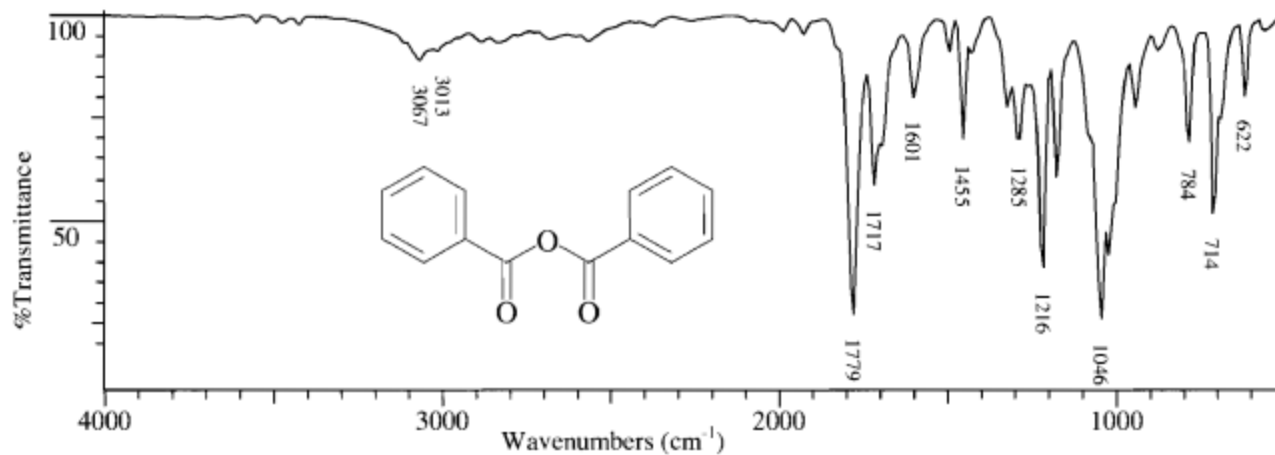
ESTERS

Strong-intensity absorptions near $1300\text{--}1000\text{ cm}^{-1}$.



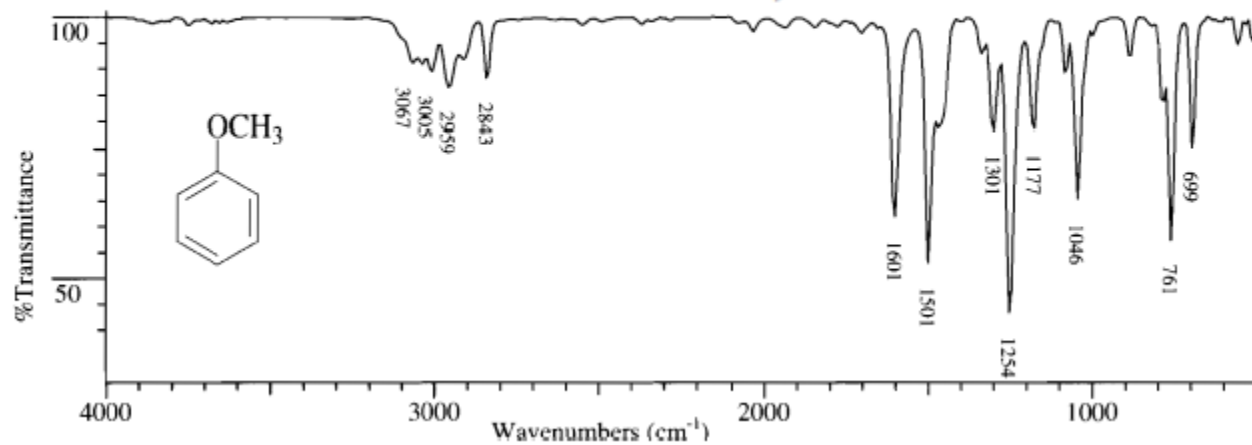
ANHYDRIDES

Two C=O absorptions near 1810 and 1760 cm⁻¹.



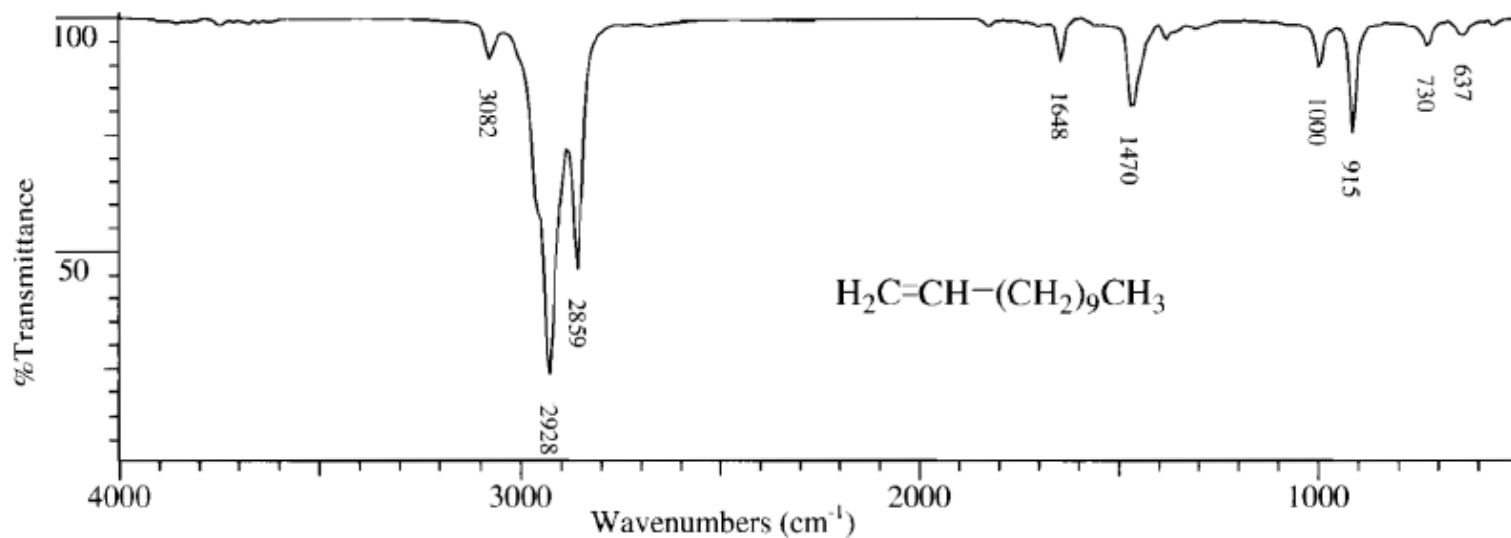
ETHERS

Check for C—O near $1300\text{--}1000\text{ cm}^{-1}$ (and absence of O—H near 3400 cm^{-1}).



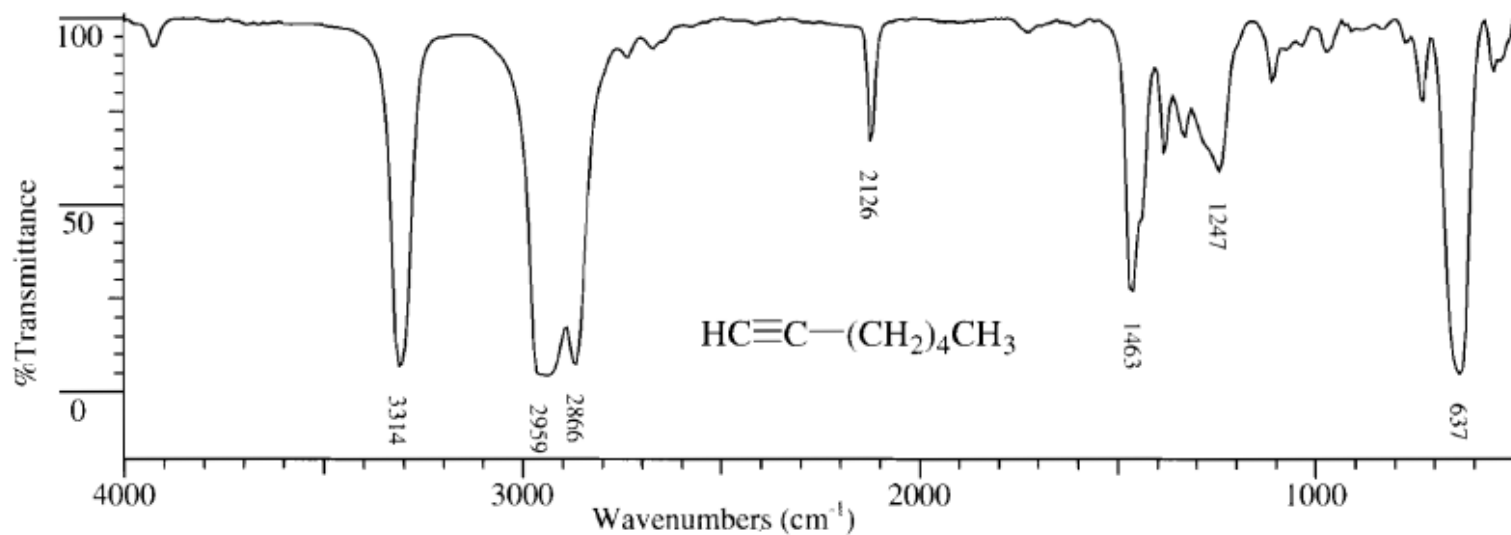
DOUBLE BONDS AND/OR AROMATIC RINGS

- C=C is a weak absorption near 1650 cm.



TRIPLE BOND

- $\text{C}\equiv\text{C}$ is a weak, sharp absorption near 2150 cm^{-1} .
- Check also for acetylenic $\text{C}-\text{H}$ near 3300 cm^{-1} .



AROMATIC COMPOUNDS

- - The C-C bond stretching gives a set of characteristic sharp peaks between 1450-1600 cm^{-1} .
- Substitution pattern can be analyzed by peaks at 600-800 cm^{-1} .



Example: Methyl benzene

