

Reminder: These notes are meant to supplement, not replace, the laboratory manual.

Infrared Spectroscopy

History and Application:

Infrared radiation is simply one segment of the electromagnetic spectrum of which visible light is a part. Human eyes do not detect infrared radiation. Infrared spectroscopy is a method to quantify how much of this type of radiation is absorbed by sample materials. Different chemical functional groups absorb specific wavelengths of radiation in the IR range. By examining the characteristic absorbance patterns of a compound, the functional groups present can be determined.

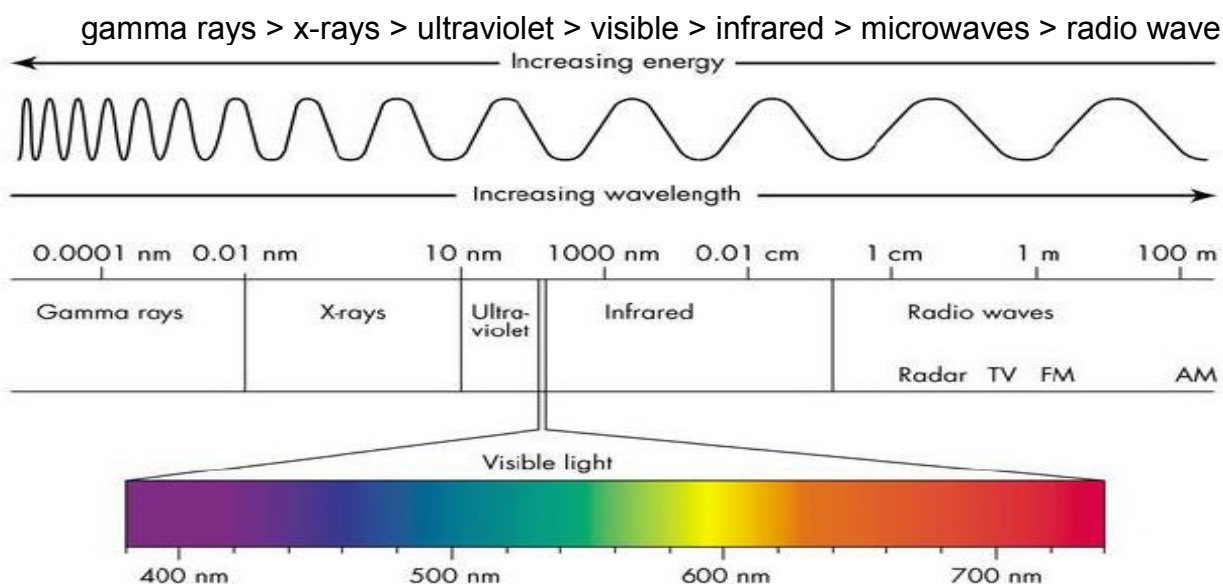
The first commercial Infrared Spectrometer was offered for sale by Perkin-Elmer in 1944.ⁱ These early analyses were very labor intensive exercises. The first computer controlled IR was also developed by Perkin-Elmer in 1976.ⁱ The organic labs have four advanced Perkin-Elmer Fourier Transform Infrared (FT-IR) machines that you will use today. Each of these machines cost approximately \$25,000. These machines obtain high quality spectra rapidly and are easy to use.

IR is a reliable and facile analytical technique that has many real world applications. IR is used in many food and chemical production facilities to measure product qualityⁱⁱ ranging from measuring the fat and moisture in meat to film thickness of polymer sheets.

Laboratory Application

Each of you will receive an unknown compound. You will analyze it on one of the four IR spectrometers and obtain an IR spectrum. Your careful analysis of the spectrum will allow you to correctly identify your unknown material.

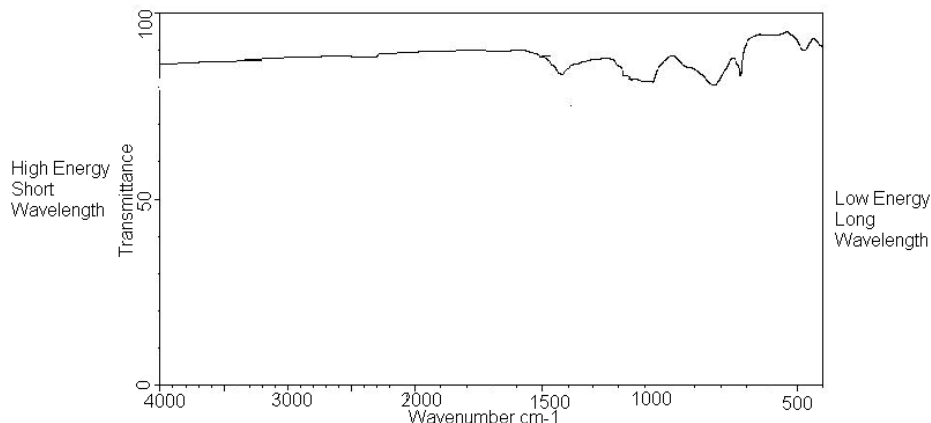
1. Here is a list of the types of electromagnetic radiation, in order of decreasing energy per photon.



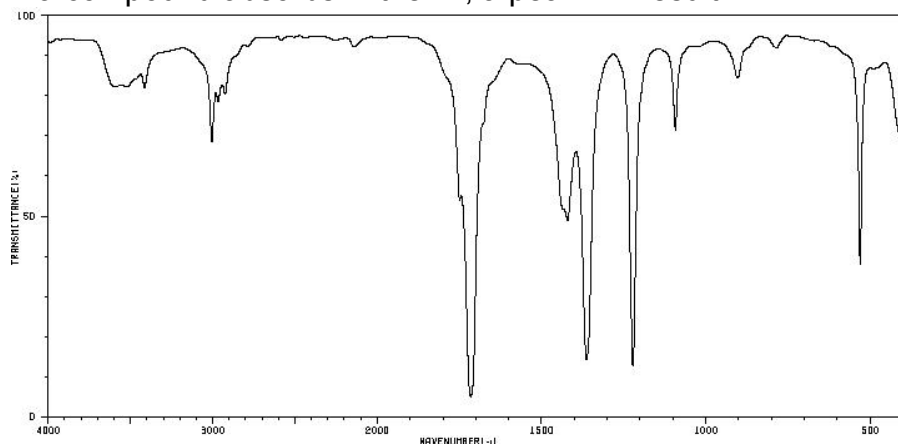
In the equation $E = h \nu = hc / \lambda$, E is the energy, h is Planck's constant, ν is the frequency, c is the velocity of light, and λ (lambda) is the wavelength. This means that $E \propto \nu \propto \frac{1}{\lambda}$, or that Energy is proportional to frequency and frequency is proportional to the inverse of wavelength. In other words as energy increases, frequency increases, and wavelength decreases.

2. Different chemical bonds absorb different energy photons of infrared (IR) radiation. The absorbed energy causes the bond to vibrate, stretch or bend faster (change to a higher-energy state). The observance of the different characteristic IR energy being absorbed allows the detection of different chemical bonds within a molecule.
3. Infrared (IR) spectroscopy is the study of molecules by their absorption (and transmission) of infrared light. Chemists use IR spectroscopy to identify the functional groups present in molecules. We also compare IR spectra of unknown compounds with literature spectra of known compounds, for purposes of identification. These comparisons are most often performed using computers.
4. Different types of samples can be used when taking IR spectra.
Neat samples are liquid samples with no added solvent. (We don't call them pure samples because they can be mixtures, and often are.)
Salt plates are disks or rectangles of clear salt (typically NaCl) with parallel faces. Handle salt plates only by the edges, because fingerprints can contaminate the plates. Do not allow any water or wet solvents to contact the salt plates. Neat samples or Nujol mulls are placed between two salt plates for IR spectroscopy. To clean salt plates, rinse them with ethanol or some other organic solvent. DON'T wash them with water; the salt will dissolve.
Nujol is a brand name of mineral oil similar to baby oil. Mineral oil is distilled from petroleum, and is a mixture of alkanes having medium molecular weights (higher than kerosene, lower than paraffin). A Nujol mull is a thick suspension of a solid with the oil. Mulls are made by grinding the solid and oil together in a mortar and pestle.
KBr pellets are solid mixtures of KBr and a solid sample. To make a KBr pellet mix a ground solid sample with ground potassium bromide. Subject this mixture to high pressure to form a disc shaped pellet. This pellet will be translucent or transparent to IR light and an IR spectrum can be taken directly.
5. A typical IR spectrum is a graph. The X axis gives the frequency in units of cm^{-1} , reciprocal centimeters or wavenumbers. This is equal to the number of waves in a centimeter. As a wavelength gets shorter, more waves can fit within one centimeter, and the wavenumber increases. As wavelength decreases, wavenumber increases, and energy increases. The Y axis gives the relative amount of IR photons that are absorbed. If a sample does not absorb any

photons, a straight baseline will result close to 100 % transmittance (0 % absorbance). The baseline is at the upper edge of the spectrum.

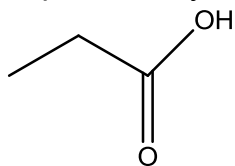


If a compound absorbs in the IR, a peak will result.

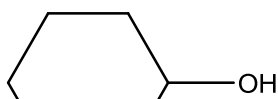


The compound which was responsible for the above spectrum has a number of characteristic absorbancesⁱⁱⁱ. The strongest absorbance is at 1720 cm^{-1} . You will soon learn to see that this particular absorbance is characteristic of carbonyl groups.

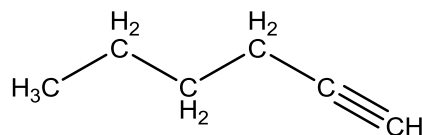
5. Below are 9 possible compounds that may be used as unknowns. There are no actual chemical reactions for this experiment. Write all of the structures and names of the compounds in your pre-lab write up.



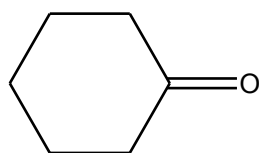
Propanoic Acid



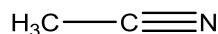
Cyclohexanol



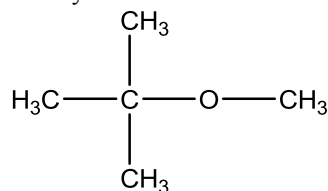
1-Hexyne



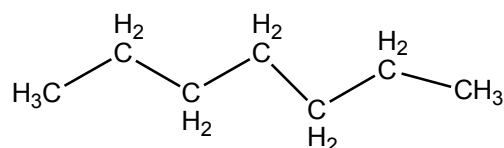
Cyclohexanone



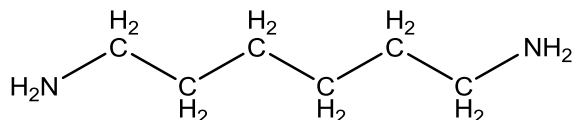
Acetonitrile



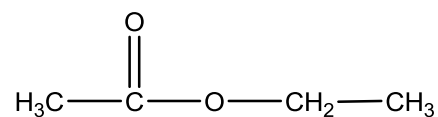
2-methoxy-2-methyl propane (MTBE)



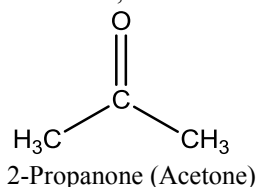
Heptane



1,6-Diaminohexane



Ethyl Acetate



2-Propanone (Acetone)

6. Safety considerations:

All of the compounds you will take IR spectra of in this experiment are at least slightly toxic and flammable. Wash your hands after conducting the experiment.

Infrared radiation is relatively (but not completely) harmless. The safety instructions on the IR spectrometer say “Do not stare into beam,” because long exposures can be damaging.

7. Read over and print out a paper copy of “Sample IR Spectra” found on the Chemistry department organic web site. Bring this with you to lab. Your instructor will go over methods to properly and easily interpret IR spectra. This information will be used to determine the identity of your unknown material.

REFERENCES

ⁱ Perkin-Elmer History of IR

http://las.perkinelmer.com/content/RelatedMaterials/Brochures/BRO_60YearsInfraredSpectroscopy.pdf (December 26, 2010)

ⁱⁱ NDC-Online Measurement Company description of applications <http://www.ndcinfrared.com/NDC/> (September 7, 2011)

ⁱⁱⁱ Spectra Database, SDBSWeb : <http://riodb01.ibase.aist.go.jp/sdbs/> (National Institute of Advanced Industrial Science and Technology), (December 26, 2010)