

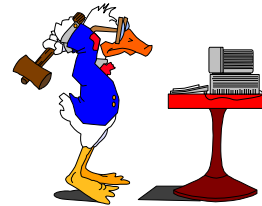
Pharmacy 325

Infrared (IR) Spectroscopy

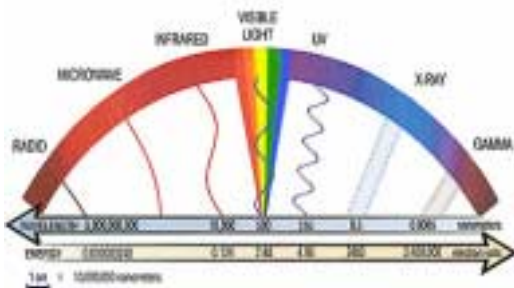
Dr. David Wishart
Rm. 2123
Ph. 492-0383
david.wishart@ualberta.ca
Hours: anytime after 4 pm

Lecture Notes Available At:

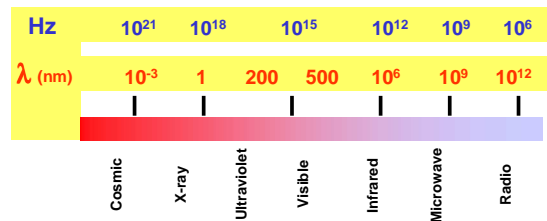
- <http://redpoll.pharmacy.ualberta.ca>
- <http://www.pharmacy.ualberta.ca/pharm325/>



Electromagnetic Spectrum



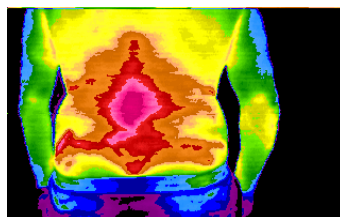
Electromagnetic Spectrum



Different Types of IR

- **Near Infrared**
 - 1-3 μ (wavelength) used in particle and composition analysis
- **Middle Infrared**
 - 3-50 μ (wavelength) primarily used in compound characterization
- **Far Infrared**
 - 50-1000 μ (wavelength) not used

IR in Everyday Life



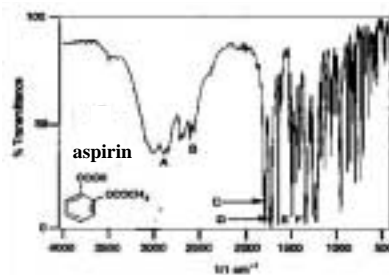
Thermal Imaging (Thermography)

IR in Everyday Life



Night Vision Goggles

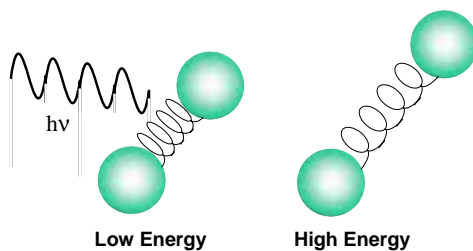
IR Spectroscopy



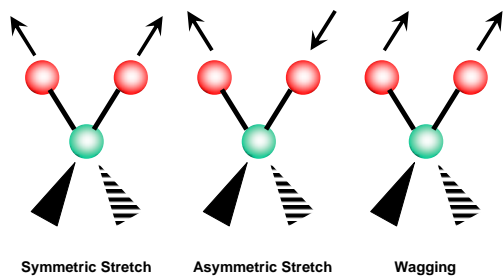
Principles of IR

- Measures heat or thermal energy in a molecule or a system
- IR spectroscopy measures the absorption of light due to bond stretching or bending
- IR energies correspond to the energies of bond stretching in most molecules
- Different types of bonds absorb at different energies (frequencies)

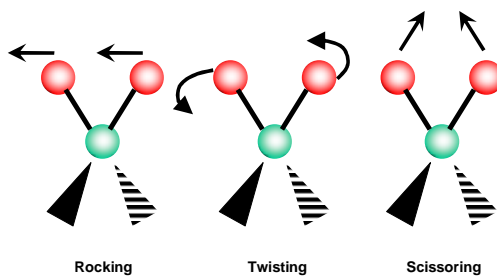
Principles of IR



Different Types of Vibration

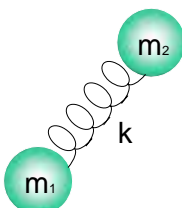


Different Types of Vibration



The IR Equation

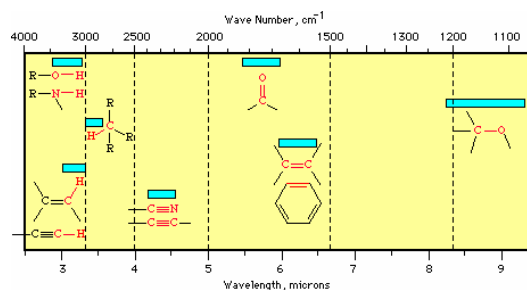
$$h\nu = \sqrt{\frac{k(m_1 + m_2)}{m_1 m_2}}$$



V_{obs} O-H > N-H > C-H > C-O > C-C > C-F

V_{obs} C≡C > C=C > C-C

IR Characteristic Vibrations



IR Units of Measurement

- **Energies** ~ 1-10 kcal/mol or 0.1 - 1.0 eV
- **Wavelength** ~ 1000 nm - 100,000 nm
- **Frequency** ~ 10¹² - 10¹⁵ Hz
- **Wave number** (1/λ) ~ 200 - 4000 cm⁻¹

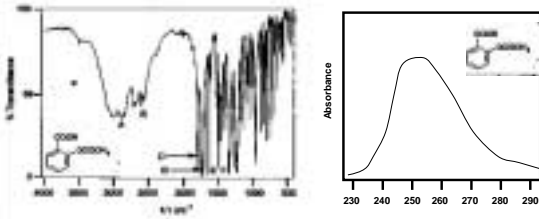
$$\nu = c/\lambda$$

Frequency is proportional to wave number

UV vs. IR

- IR has narrower peaks relative to UV
- IR yields more information than UV
- IR allows you to collect data on solids, liquids and gases
- UV is more quantitative than IR
- UV spectra are easier/faster to collect
- UV samples are easier to prepare
- UV spectrometers are cheaper

IR vs. UV

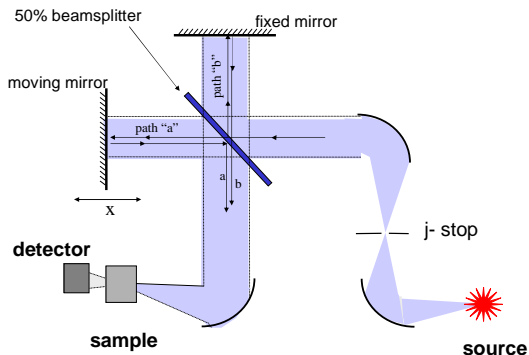


A Modern FTIR instrument

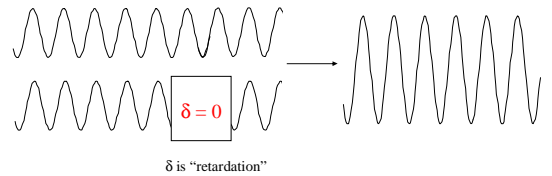


FT = Fourier Transform

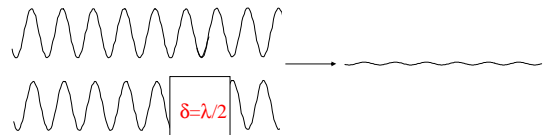
FTIR Schematic



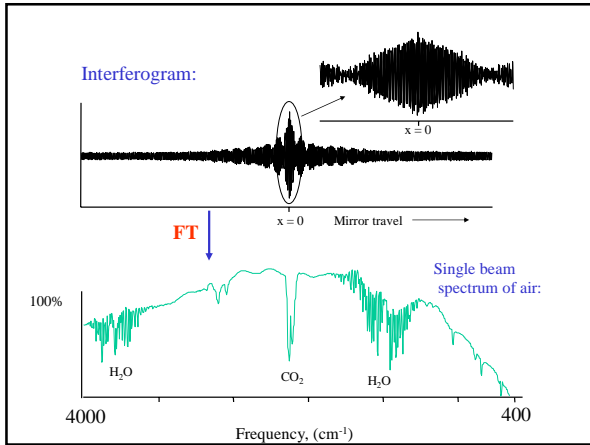
for $\delta = m\lambda$, get constructive interference



δ is "retardation"



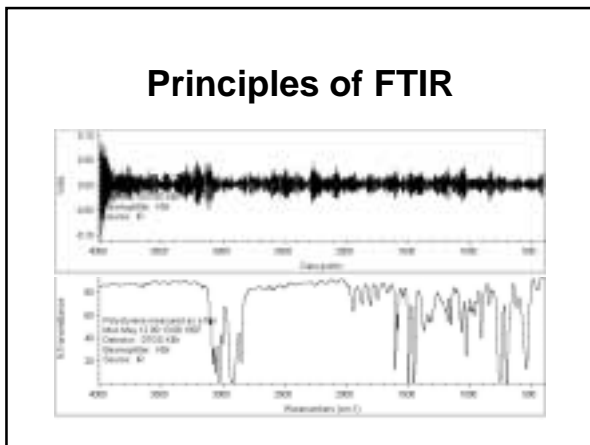
for $\delta = (m+1/2)\lambda$, get destructive interference



Fourier Transformation

$$F(\omega) = \int f(t)e^{i\omega t} dt$$

Converts from units of time to units of frequency



- ## IR Sample Preparation
- Most flexible system for analyzing all 3 states of matter (solid, liquid, gas)
 - “Neat” (analysis of liquids/oils)
 - Pellet (analysis of solids)
 - Thin Cell (analysis of dissolved solid samples - solutions)
 - Long Cell (analysis of gases)

Preparing a "Neat" IR Sample



Preparing a KBr Disk



Apparatus for KBr Disk



Pressed Disk Preparation

- Use powdered, dry KBr, KI, CsI
- Mix reagent with KBr in 1:10 ratio
- Grind material to $2\ \mu$ diameter using agate mortar or vibrating ball mill (Wig-L-Bug amalgamator)
- Place into die and press to 30 tons/in² using hand press or wrench + nut
- Remove carefully, handle with gloves

IR Liquid Sample Cell



IR Gas Sample "Cell"



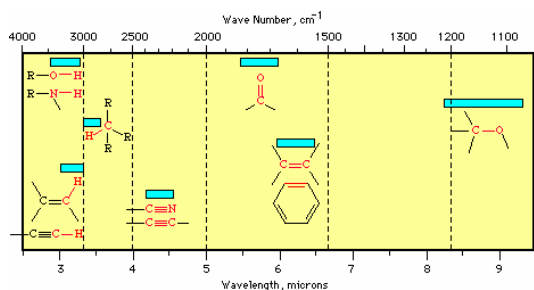
Analyzing IR Spectra

- Look for C=O peak (1820-1660 cm^{-1})
- If C=O check for OH (3400-2400 cm^{-1})
 - indicates carboxylic acid
- If C=O check for NH (3500 cm^{-1})
 - indicates amide
- If C=O check for C-O (1300-1000 cm^{-1})
 - indicates ester
- If no OH, NH or C-O then ketone

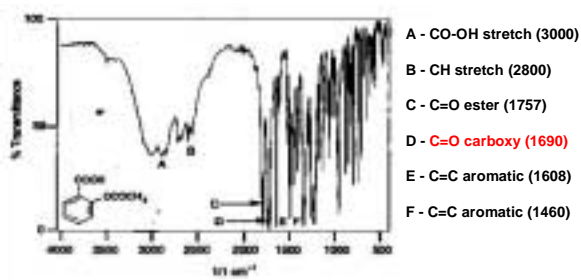
Analyzing IR Spectra

- If no C=O check for OH (3600-3300 cm^{-1})
 - indicates alcohol
- If no C=O check for NH (3500 cm^{-1})
 - indicates amine
- If no C=O & no OH check C-O (1300 cm^{-1})
 - indicates ether
- Look for C=C (1650-1450 cm^{-1}) then aromatic

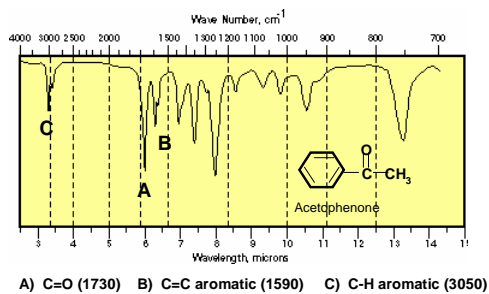
IR Characteristic Vibrations



Sample IR Spectrum #1



Sample IR Spectrum #2



Applications

- Qualitative “fingerprint” check for identification of drugs
- Used for screening compounds and rapid identification of C=O groups
- Can be used to characterize samples in solid states (creams and tablets)
- Can detect different crystal isoforms (polymorphs)
- Water content measurement

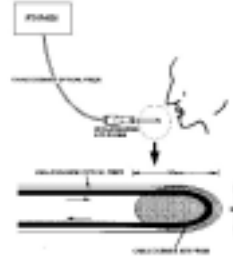
Applications

- Analysis of urine and other biofluids (urea, creatinine, protein)



Applications

- Used in non-invasive measurement of glucose



Applications of Near IR (NIR)

- Quality control of pharmaceutical formulations
- Determination of particle size
- Determination of blend uniformity
- Determination or identification of polymorphic drugs