Fourier Transform Infrared Spectrometer (FTIR)

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Components

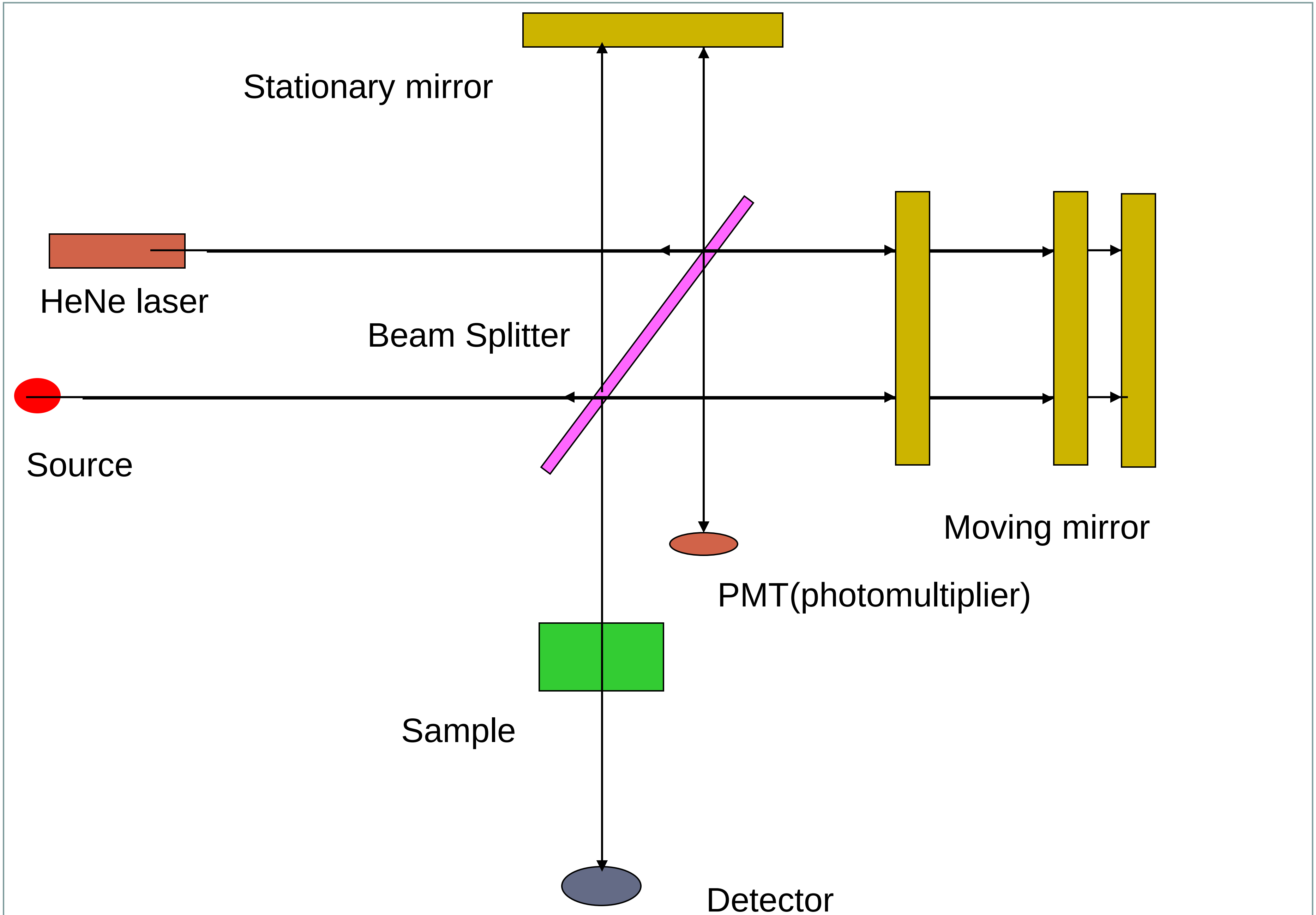
1. Source
2. Michelson Interferometer
3. Sample
4. Detector

1-Sources

- Black body radiators
- resistively heated to 1500-2200 K
- Max radiation between 5000-5900 cm^{-1} (2-1.7 μm).
- SiC rod
- CO_2 laser
- (Far IR), Tungsten filament (Near IR)

2-Michaelson Interferometer

- Beam splitter
- Stationary mirror
- Moving mirror at constant velocity
- Motor Micrometer screw
- He/Ne laser


Stationary mirror
HeNe laser
Beam Splitter
Source
Moving mirror
PMT(photomultiplier)
Sample
Detector

3- Sample

- Sample holder must be obvious to IR- salts
- Liquids
  - Salt Plates
  - 1 drop
  - Samples dissolved in volatile solvents 0.1-10%
- Solids
  - KBr pellets
  - Mulling (dispersions)
- Quantitative analysis-conserved cell with NaCl/NaBr/KBr windows

4- Detector

- Thermal transducer:

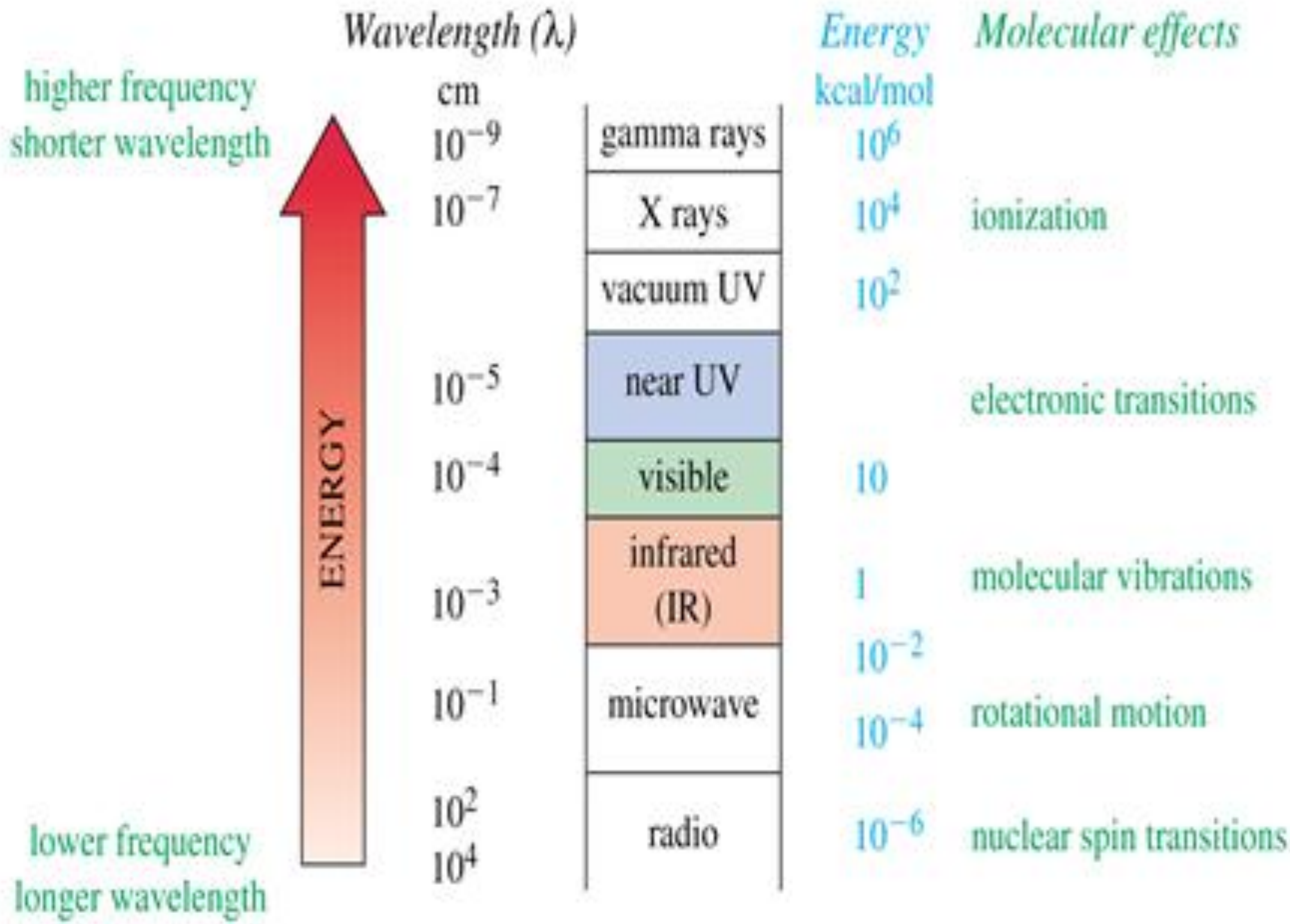
  black body, small, very low heat capacity- $\Delta T = 10^{-3}$ K, held in vacuum, signal is cut

- Thermocouples
  - Two junctions of metals, Indium and bismuth(Be)
  - One is IR detector, one is reference detector

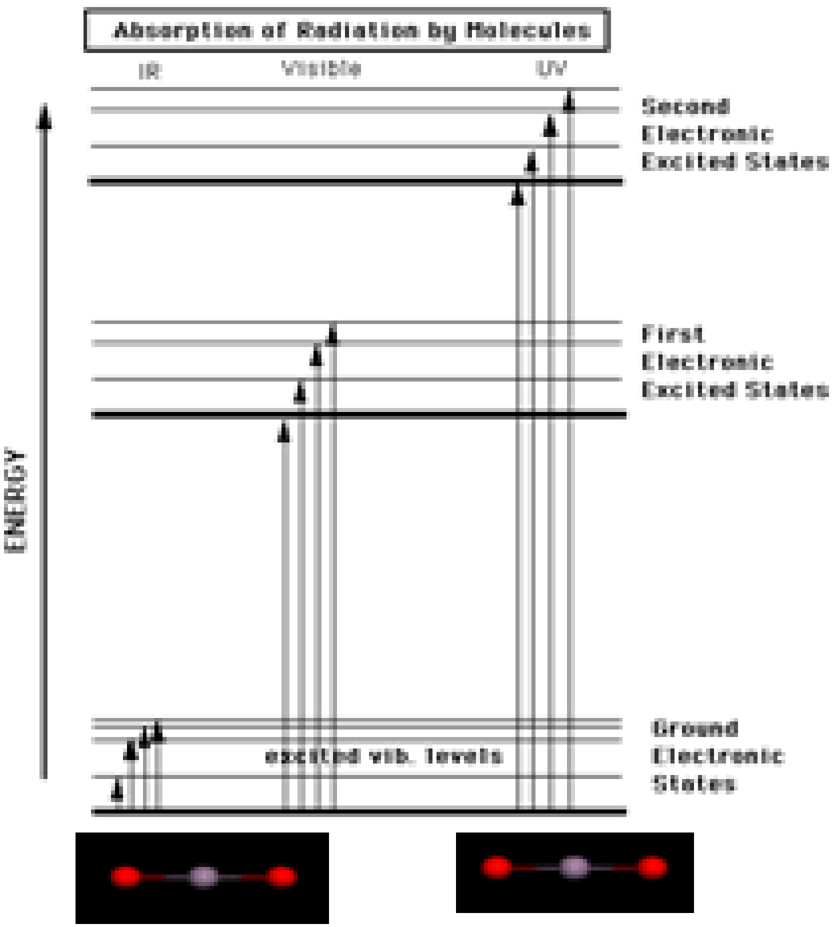
Infrared Spectroscopy (IR or FTIR)

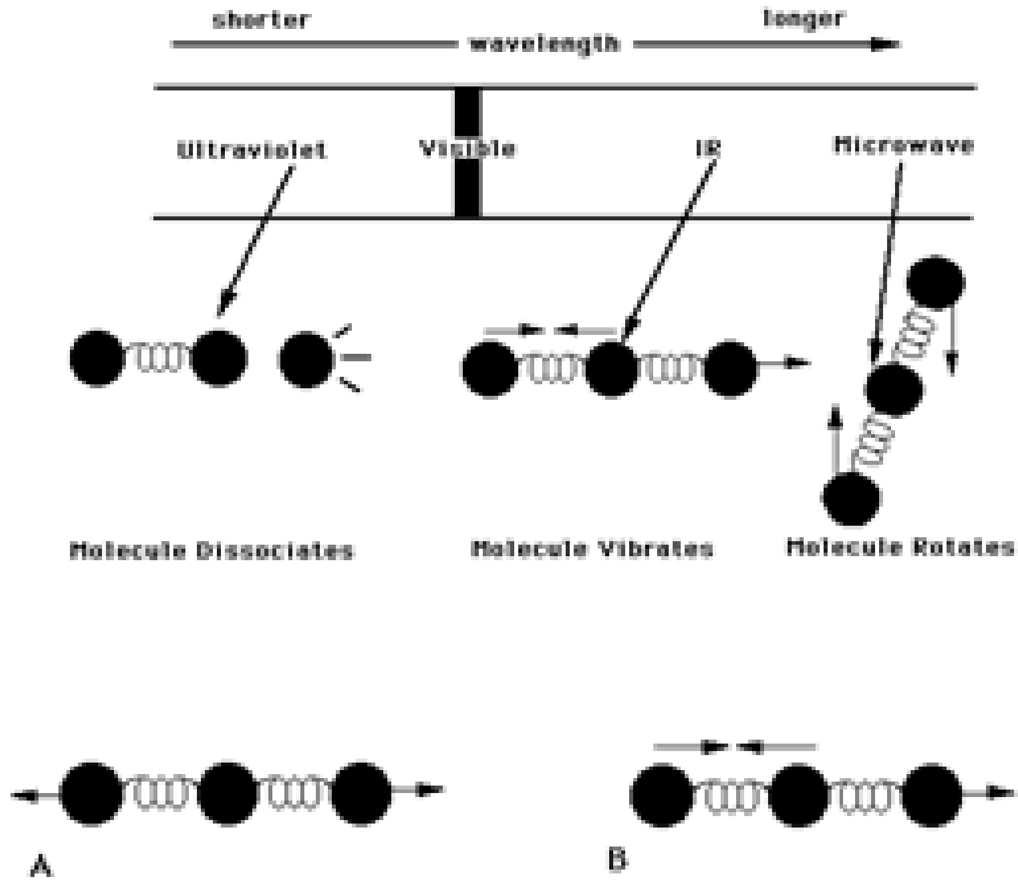
Measures the absorption of electromagnetic radiation by molecules. When molecules absorb infrared radiation of a certain energy, their bonds vibrate (stretch, contract, bend, wiggle, twist, etc.).

We can measure how much energy is absorbed at each particular wavelength and make inferences about the types of bonds that are present in the molecule.

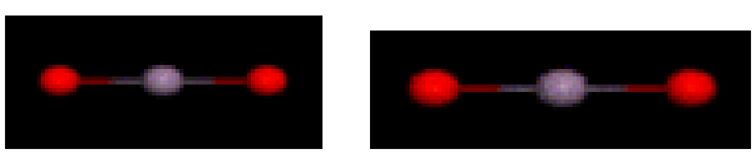

Wavelength (λ)
Energy
Molecular effects
higher frequency
shorter wavelength
cm
kcal/mol
10−9
gamma rays
10^6
10−7
X rays
10^4
ionization
vacuum UV
10^2
10−5
near UV
electronic transitions
10−4
visible
10
ENERGY
infrared
(IR)
10−3
1
molecular vibrations
10−2
10−1
microwave
10−4
rotational motion
10^2
10^4
radio
10−6
nuclear spin transitions
lower frequency
longer wavelength

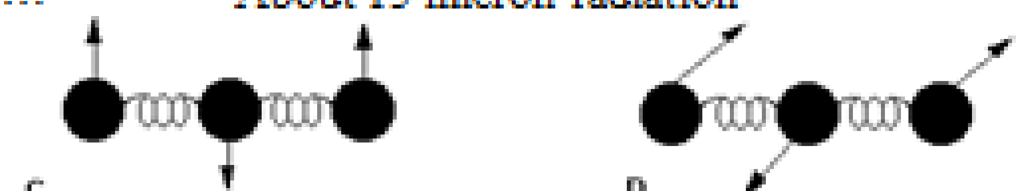
Energy Levels: Basic Ideas





Basic Global Warming: The C02 dance … About 15 micron radiation



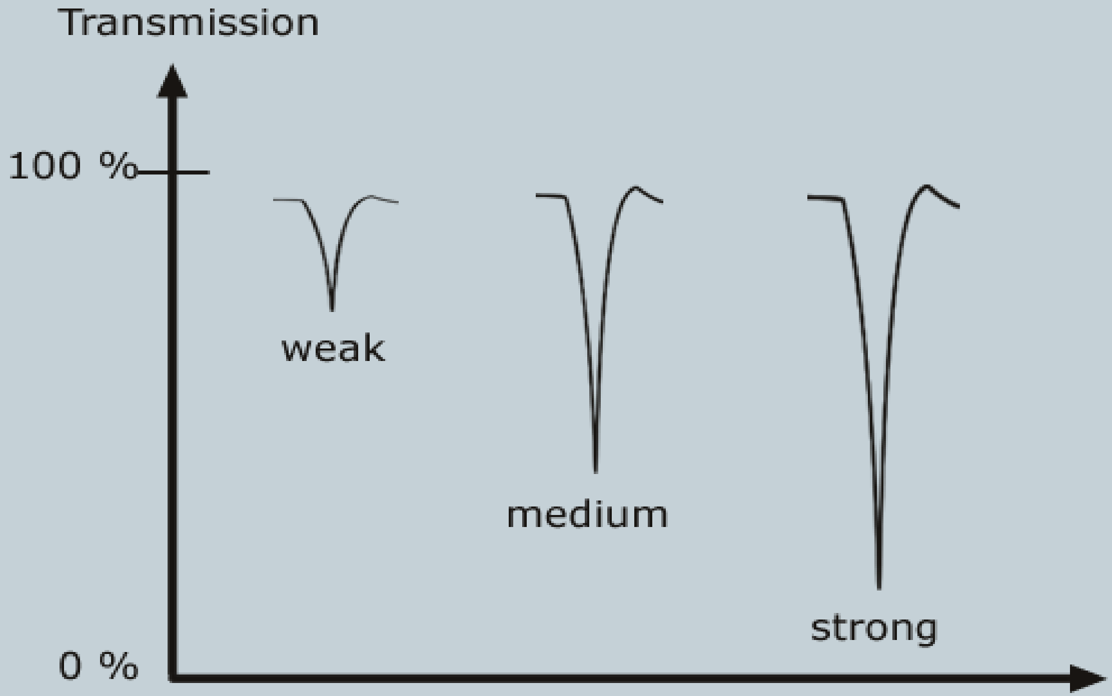


Advantages of FTIR

- Non-destructive analysis technique
- Rapid
- open to solids, liquids, gases, solutions, and rough solids
- open to organics and inorganics, though primarily used for organics
- Provides a positive (Category A) test

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.

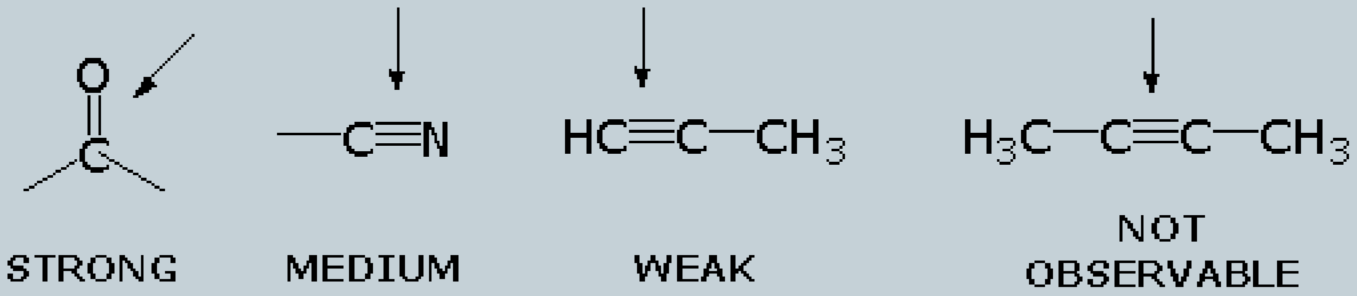


ACTIVE BONDS

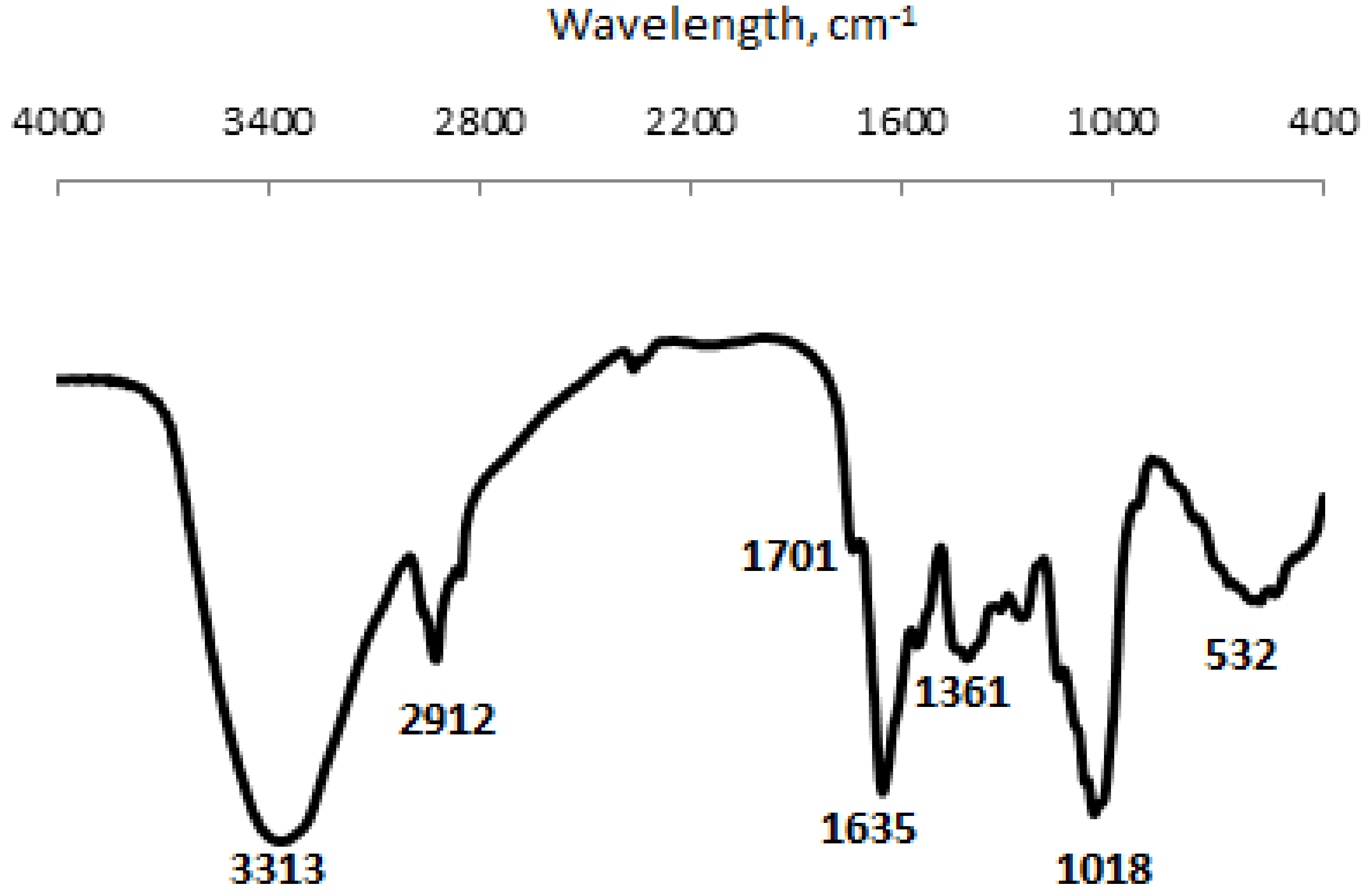
Not all covalent bonds display bands in the IR spectrum. **Only polar bonds do so. These are referred to as IR active**.

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

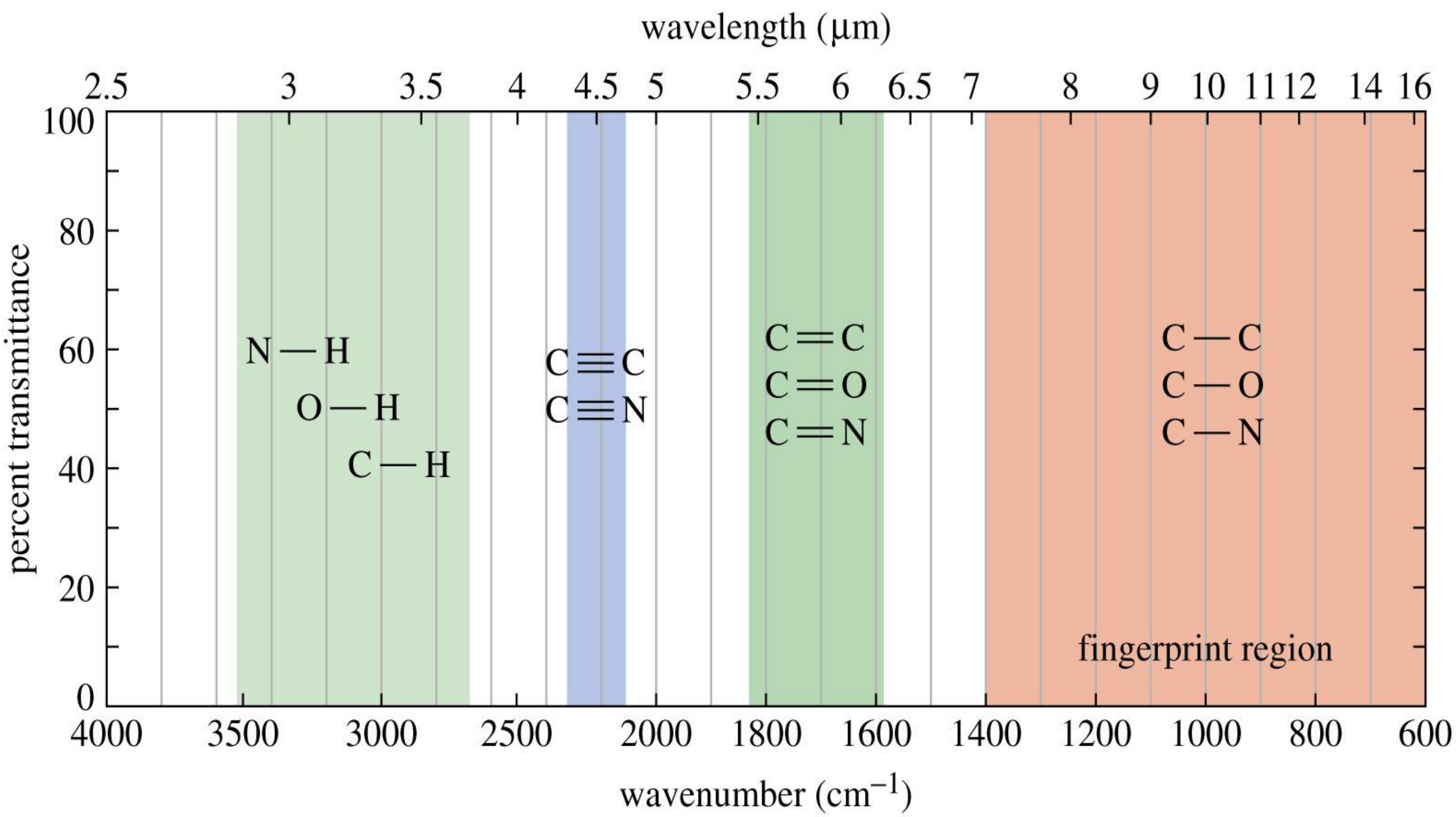
- Strongly polar bonds such as carbonyl groups (C=O) produce strong bands.
- Medium polarity bonds and asymmetric bonds produce medium bands.
- Weakly polar bond and symmetric bonds produce weak or non observable bands.



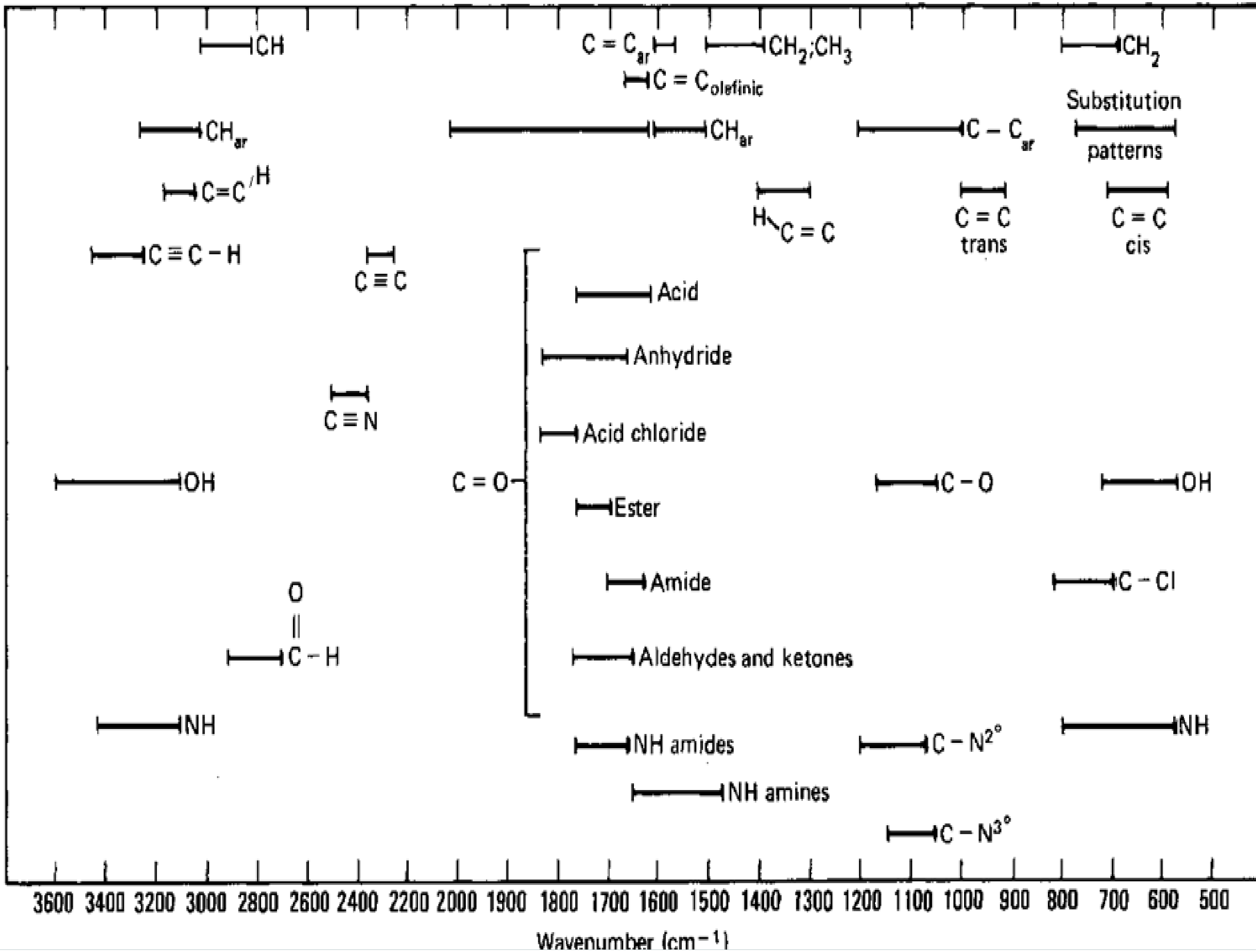
SILVER NANO PARTICLES



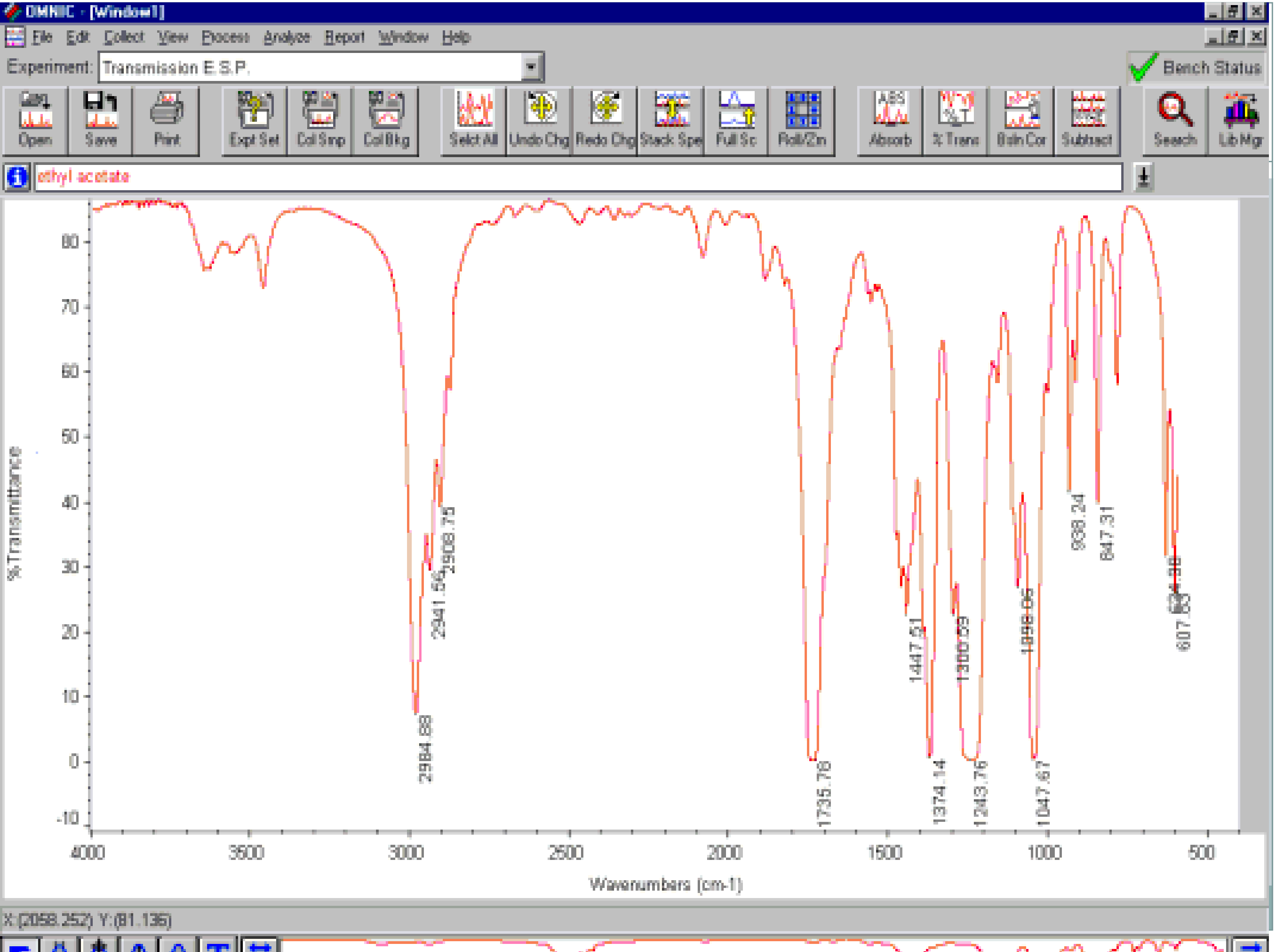
Functional Group Absorption Ranges

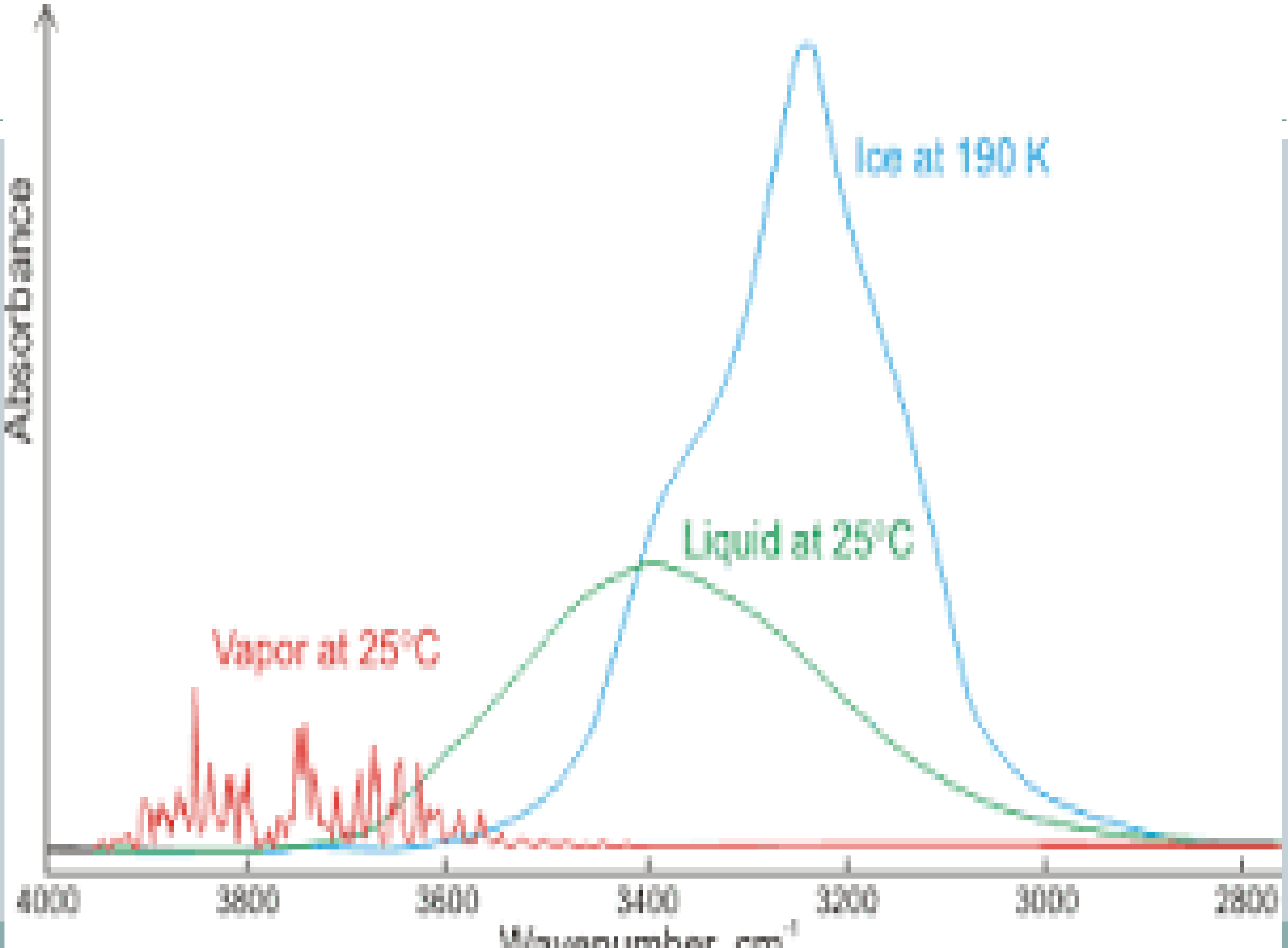


Approximate infrared absorption frequencies of various groups



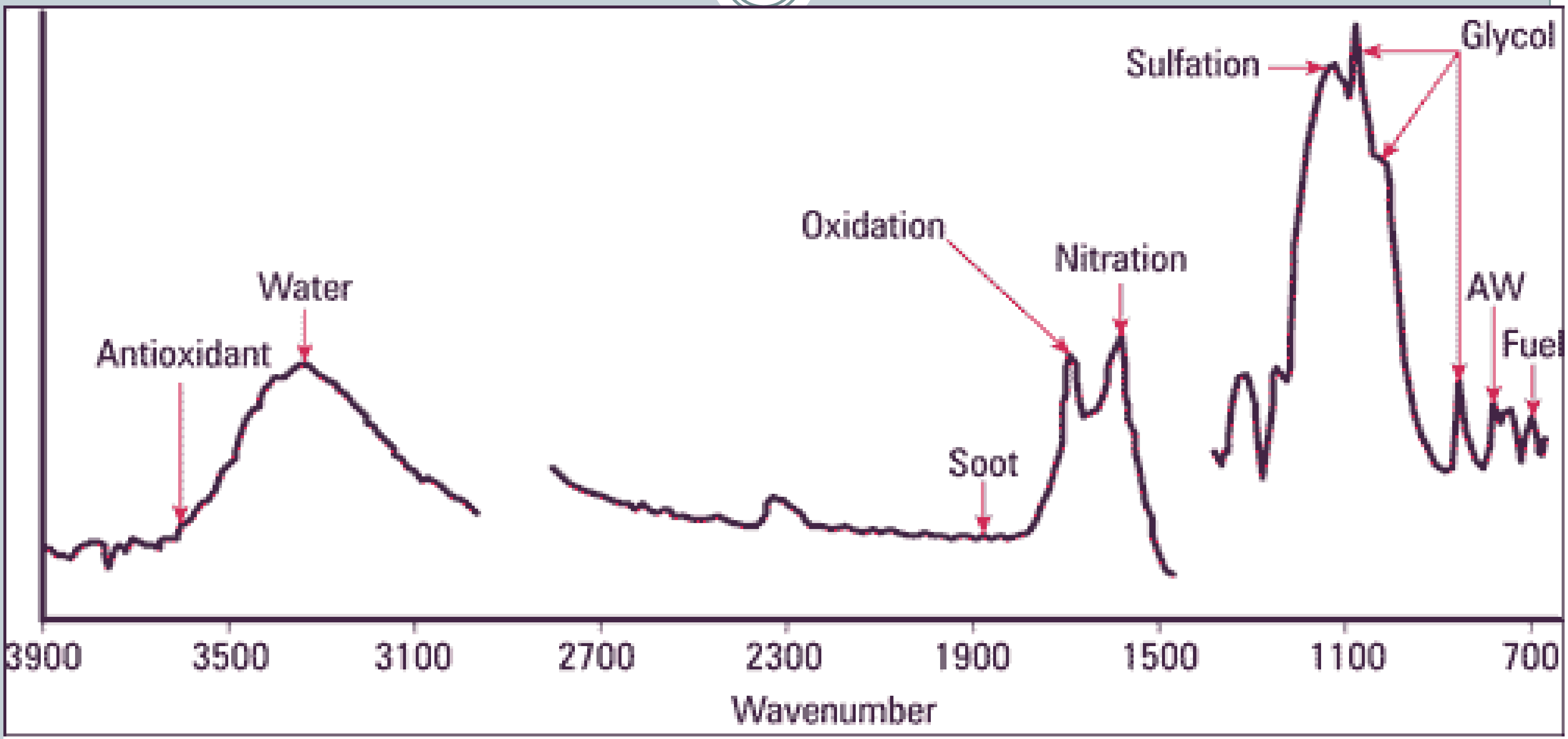
Bond	Frequency (Wavenumber Range, cm^{-1})	Intensity
C=O	1735-1680	strong
C=C	1680-1620	variable
C≡C	2260-2100	variable
C≡N	2260-2220	variable
C-H	3300-2700	variable
N-H	3150-2500	moderate
O-H	3650-3200	broad

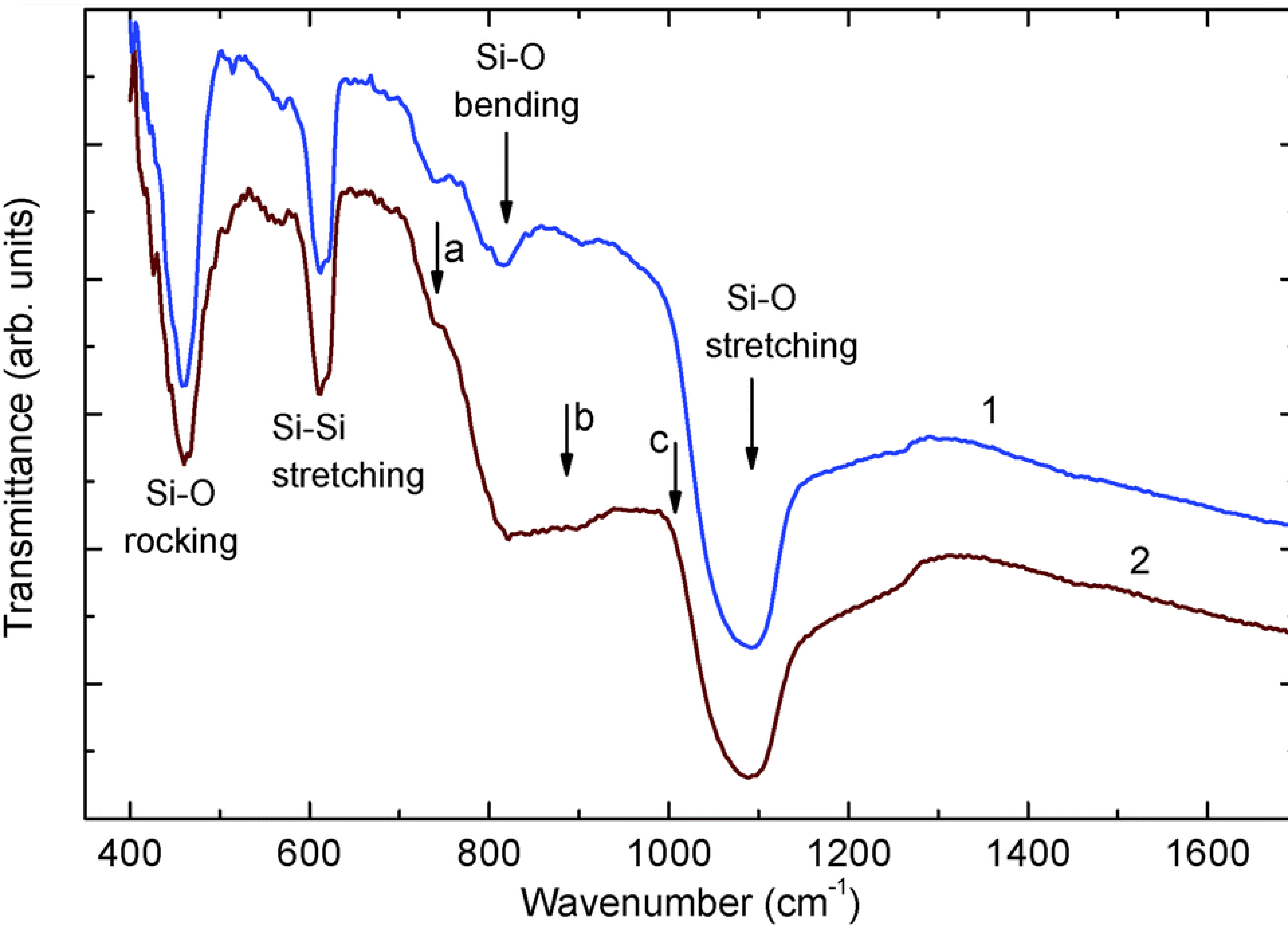

OMNIC - [Window1]
File Edit Collect View Process Analyze Report Window Help
Experiment: Transmission E.S.P.
Bench Status
Open
Save
Print
Expt Set
Col Smp
Col Bkg
Selct All
Undo Chg
Redo Chg
Stack Spe
Full Sc
Roll/Zm
Absorb
% Trans
Bsln Cor
Subtract
Search
Lib Mgr
ethyl acetate
%Transmittance
Wavenumbers (cm-1)
2984.88
2941.56
2908.75
1735.78
1447.51
1374.14
1300.59
1243.76
1098.05
1047.67
938.24
847.31
607.63
X:(2058.252) Y:(81.136)

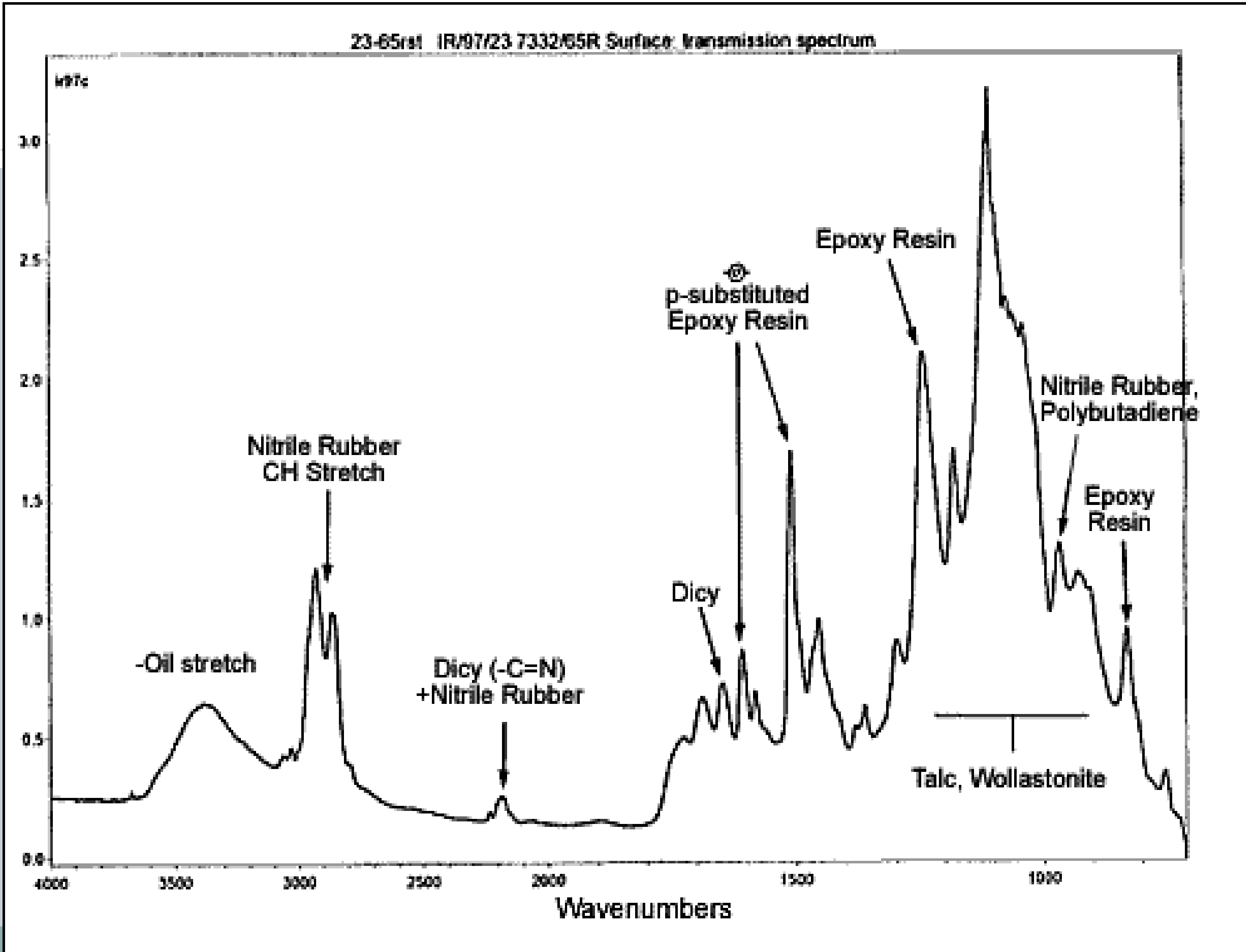


Absorbance
Ice at 190 K
Liquid at 25°C
Vapor at 25°C
4000
3800
3600
3400
3200
3000
2800
Wavenumber, cm⁻¹

OIL FTIR SPECTRUM




Transmittance (arb. units)
Wavenumber (cm^{-1})
400
600
800
1000
1200
1400
1600
Si-O
rocking
Si-Si
stretching
Si-O
bending
a
b
c
Si-O
stretching
1
2


23-65rst IR/97/23 7332/65R Surface transmission spectrum
-Oil stretch
Nitrile Rubber
CH Stretch
Dicy (-C=N)
+Nitrile Rubber
Dicy
p-substituted
Epoxy Resin
Epoxy Resin
Nitrile Rubber,
Polybutadiene
Epoxy
Resin
Talc, Wollastonite
4000
3500
3000
2500
2000
1500
1000
Wavenumbers
0.0
0.5
1.0
1.5
2.0
2.5
3.0

Wavelength and Wavenumber

- Wavelength = 1 / Wavenumber
- For the IR, wavelength is in microns.
- Wavenumber is typically in 1/cm, or cm^{-1}.
- 5 microns corresponds to 2000 cm^{-1}.
- 20 microns corresponds to 500 cm^{-1}.

- **15 microns corresponds to 667 cm^{-1}. Much ‘worldly’ IR energy at the wavenumber.**

Fourier Transforms cont.

- The Continuous Fourier Transform, for use on continuous signals, is defined as follows:

$$F(w) = \int_{-\infty}^{\infty} f(x)\, e^{(-2\pi w i x)}\, dx$$

- And the Inverse Continuous Fourier Transform, which allows you to go from the spectrum back to the signal, is defined as:

$$f(x) = \int_{-\infty}^{\infty} F(w)\, e^{(2\pi w i x)}\, dw$$

F(w) is the spectrum, where w represents the frequency, and f(x) is the signal in the time where x represents the time. i is sqrt(-1)


Thermo