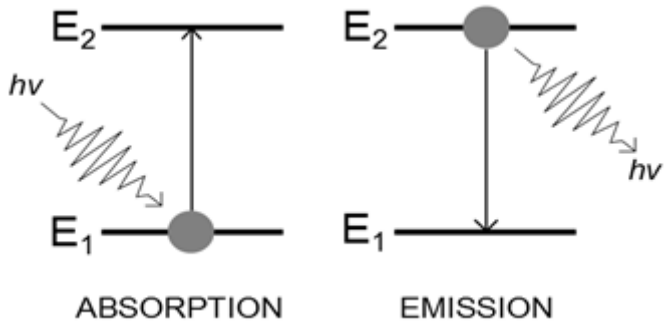
Unit IV: Instrumental Methods and Applications

Introduction to spectroscopy–types of energy present in molecules, types of spectra, UV-Vis spectroscopy – principle, types of electronic transitions, Instrumentation and applications; Infrared spectroscopy – principle, types of vibrational modes, Instrumentation and applications; working principle and applications of SEM, TEM, and XRD.

The transitions of the electron from one energy level to another energy level in an atom results the emission or absorption spectrum and energy difference between these energy levels gives spectral lines in different regions. The resulted atomic spectrum provides useful information. Similarly molecular spectra provide useful information regarding the molecular structure. The energy transferred in the form of electromagnetic radiation.



Spectroscopy is the study of interaction of matter and electromagnetic radiation.

TYPES OF ENERGY PRESENT IN MOLECULES

If E is the energy of a molecule, it can be expressed as: $\mathbf{E = (E_{trans}) + (E_{rot}) + (E_{vib}) + (E_{ele})}$

i. **Translational Energy ($\mathbf{E_{trans}}$)** is concerned with the overall movement of the molecules along the three axes. It is significant only in gases and to a lesser extent for liquids.
ii. **Rotational energy ($\mathbf{E_{rot}}$)** involves the spinning of molecules about the axes passing through their center of gravity.
iii. **Vibrational energy ($\mathbf{E_{vib}}$)** is associated with vibrations within a molecule such as the stretching or the bending of bonds.
iv. **Electronic energy ($\mathbf{E_{ele}}$)** involves promotion of electrons to higher levels on absorption of energy

The magnitude of the various forms of these energies and the difference in energy levels vary considerably. .....

Difference between the energy levels for bringing the specific type:

- Electronic energy **($\mathbf{E_{ele}}$)**change = About 100 – 10^4 kJ / mol
- Vibrational energy**($\mathbf{E_{vib}}$)** change = About 1 – 100 kJ / mol
- Rotational energy **($\mathbf{E_{rot}}$)**change = About 0.01 – 1 kJ / mol

TYPES OF SPECTRA:

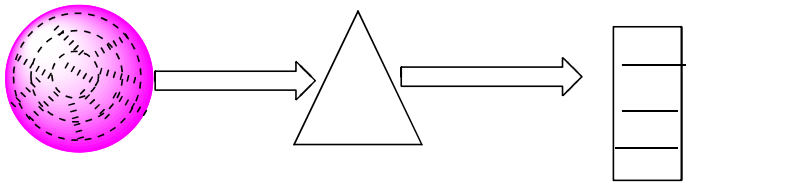
The two types of spectra can be seen in spectroscopy.

i) Emission spectra. **ii)** Absorption spectra.

(i) Emission spectra**:**

- Molecules give emission spectra when subjected to intense heat or electric discharge.
- In this situation the molecules gets excited by obtain the necessary energy and these excited states are unstable and as result, the molecules return to their lower energy state, by emitting radiation in the form of photon and the corresponding frequency is recorded as the emission spectrum.
- If the transition is from upper energy level (E_2) to lower energy level (E_1), the frequency (γ) of the emission spectrum is given by:

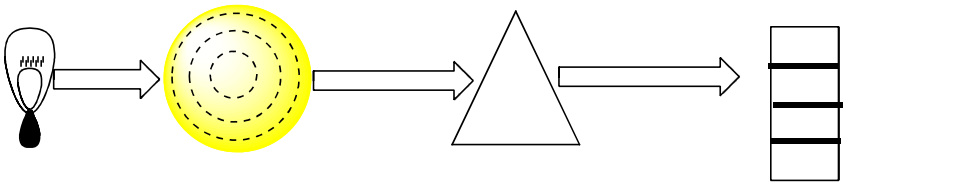
$$v = \frac{E_2 - E_1}{h} = \frac{\Delta E}{h}$$



Hot Gas **Prism** **Emission spectrum**

(ii) Absorption Spectra**:**

- When a substance is irradiated with electromagnetic radiation, the molecules may transferred from the ground state to the excited state, by absorbing the incident photons.
- This process, is known as absorption and the resultant spectrum, is known as *absorption spectrum*.



Source of Energy **cold Gas** **Prism** **Absorption spectrum**

- Energy absorption occurs only when the energy difference between the ground state and higher energy level is exactly matched by the energy of the incident electromagnetic radiation. The molecular spectra are more complicated than atomic spectra, because they range over wider regions of the electromagnetic spectrum and their interpretation is often more difficult.
- The energy absorbed (ΔE) by a molecule may bring about changes in one or more of its energy levels such as rotational, vibrational and electronic.
- Absorption spectra helps to elucidate the structure of molecules.

- The absorption spectra further divided into three different types on the basis of the radiation absorbed.
    - **a)** Microwave absorption spectra
    - **b)** Infrared absorption spectra
    - **C)** Ultraviolet and Visible absorption spectra

Ultraviolet (UV) Spectroscopy

UV spectroscopy is an important tool in analytical chemistry. The other name of UV (Ultra-Violet) spectroscopy is Electronic spectroscopy as it involves the promotion of the electrons from the ground state to the higher energy or excited state and this is the fundamental principle here. UV Spectroscopy primarily used to measure the multiple bond or aromatic conjugation with in molecules.

Principle &Instrumentation of UV-Vis Spectrophotometer

Principle of UV spectroscopy

UV spectroscopy obeys the Beer-Lambert law, which states that: ***when a beam of monochromatic light is passed through a solution of an absorbing substance the rate of decrease of intensity of radiation is directly proportional to the concentration of the substance and the path length of the light through the solution.***

The mathematical expression of Beer-Lambert law is $A = \log (I_0/I) = ECL$

A = absorbance

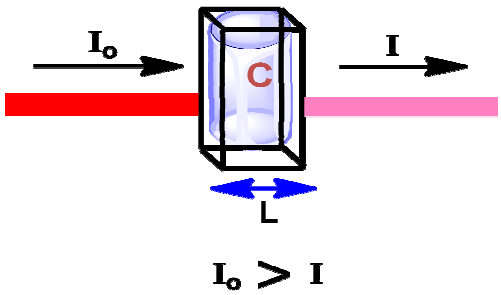
Io = intensity of light leaving reference cell

I= intensity of light leaving sample cell

C = molar concentration of solute

L = length of sample cell (cm.)

E = molar absorptivity



The absorbance of a transition depends on two external assumptions.

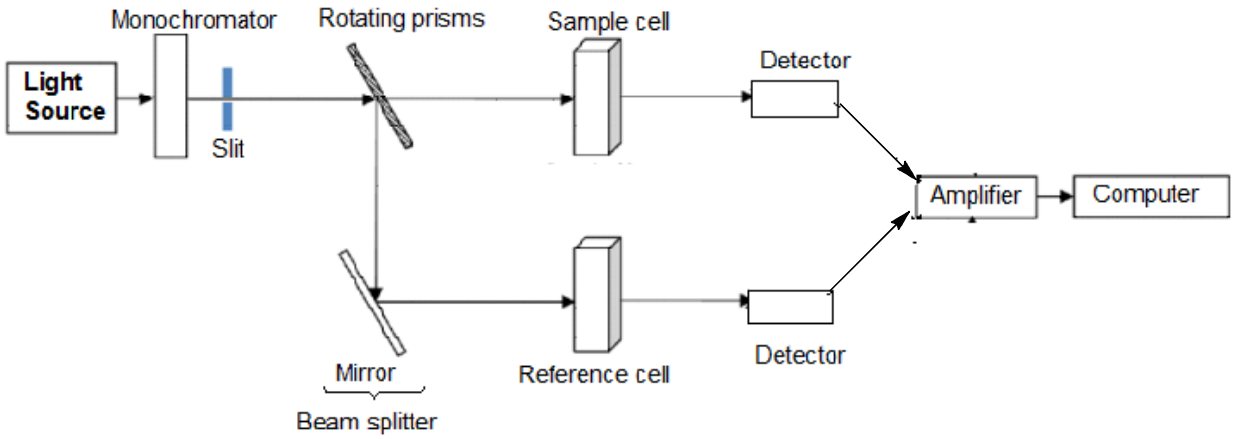
i) The absorbance is directly proportional to the concentration (c) of the solution of the sample used in the experiment.

ii) The absorbance is directly proportional to the length of the sample cell (L), which is equal to the width of the cuvette.

Molar absorptivity, is a measure of how well a chemical species absorbs a given wavelength of light. [illegible].

Instrumentation and working of UV spectroscopy

Instrumentation and working of the UV spectrometers can be studied simultaneously. Most of the modern UV spectrometers consist of the following parts.



Block diagram of uv-vis spectrophotometer

Light Source: Tungsten filament lamps and Hydrogen-Deuterium lamps are most widely used and suitable light source as they cover the whole UV region. The electrical excitation of deuterium or hydrogen at low pressure produces a continuous UV spectrum.

Monochromator: Monochromators generally composed of prisms and slits. A beam of monochromatic light is split with the help of **beam splitter** (rotating prisms) into two equal halves called as sample beam and reference beam. The most of the spectrophotometers are double beam spectrophotometers.

Sample and reference cells: The sample beam is directed through a transparent cell containing a solution of the compound being analyzed and the reference beam is directed through an identical cell that contains only the solvent. These cells are made of either silica or quartz. Glass cannot be used for the cells as it also a sorbs light in the UV region.

Detector: Generally two potocells serve the purpose of detector in UV spectroscopy. One of the photocell receives the beam from sample cell and second detector receives the beam from the reference. The intensity of the radiation from the reference cell is stronger than the beam of sample cell. This results in the generation of alternating currents in the photocells.

Amplifier: The alternating current generated in the photocells is transferred to the amplifier. Generally current generated in the photocells is of very low intensity. The amplifier is used to convert these information into a physically measurable information.

Computer / printer: The amplifier is connected to the computer to store and print the data generated for the desired compound. The instrument gives output graph contains a plot of the wavelength of the entire region versus the absorbance (A) of the light at each wavelength. Such a graph is known as an absorption spectrum.

Electronic Transitions

According to theory of electronic spectroscopy, when the molecule absorbs UV or visible light, its electros promoted to higher energy levels. i.e. From bonding to anti-bonding molecular orbitals.

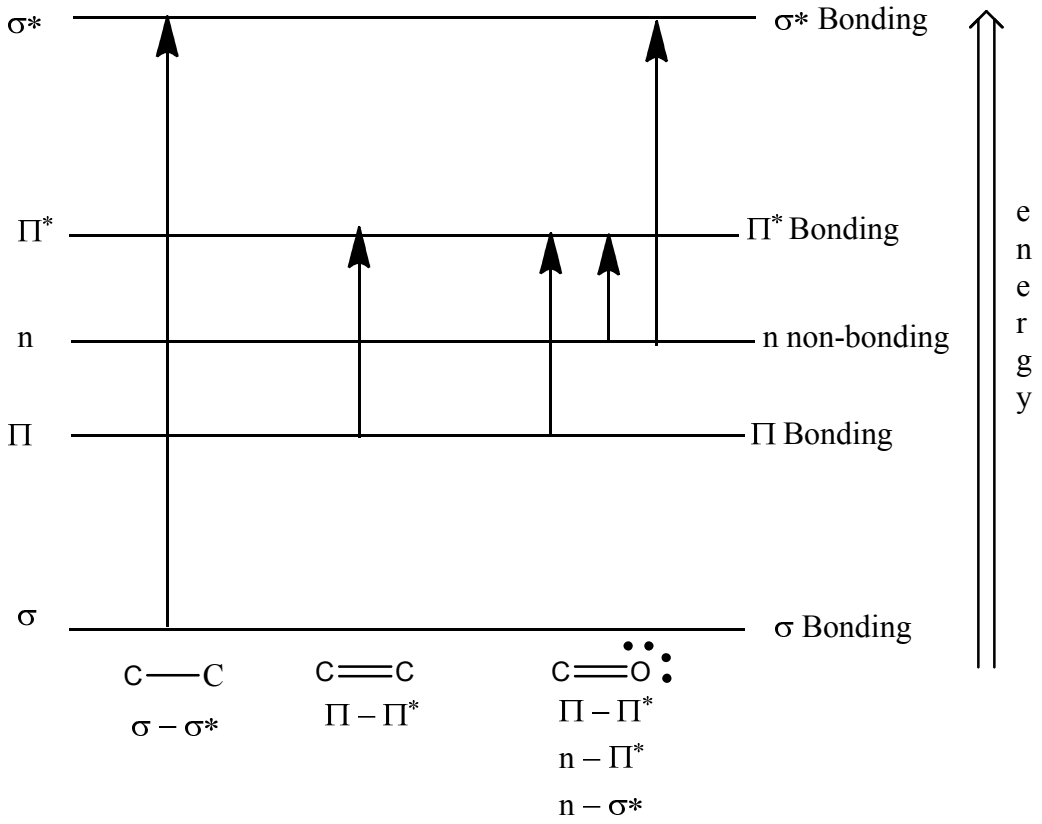


Fig: Relative energies of orbitals and possible transitions between them

Electronic energy levels σ → σ* > n → σ*>π → π* >n → π*

Energy decreases

When excitation occurs, an electron from one of the filled **σ, π** or **n** molecular orbitals get excited to vacant **σ* or π***anti-bonding molecular orbitals (shown in below fig). The **σ** electrons requires a high energy for excitation to **σ*.** It is not essential that exposure of a compound to ultraviolet or visible light must always give rise to an electronic transition

APPLICATIONS OF UV-VIS SPECTROPHOTOMETER

i) Identification of extent of conjugation:

With the increase in double bonds the absorption shifts towards the longer wavelength

ii) Detection of Impurities:

a) Additional peaks can be observed due to impurities in the sample.

b) Further the absorption of the sample solution is compared with the absorption of the reference solution. The intensity of the absorption can be used for the relative calculation of the purity of the sample substance. By also measuring the absorbance at specific wavelength, the impurities can be detected. UV absorption spectroscopy is one of the best methods for determination of impurities in organic molecules.

iii) Identification of an unknown compounds:

The spectrum of unknown compound is compared with the spectrum of a standard compound and it both the spectrums coincide then it confirms the identification of the unknown compound.

iv) **Structure elucidation of organic compounds:**

Helps in the structure elucidation of organic molecules by knowing the presence of saturated or unsaturated bonds and hetero atoms.However,UV spectroscopy is not helpful in the detection of individual functional groups

v) **Quantitative analysis:**

Used for the quantitative determination of compounds that absorb UV radiation. This determination is based on Beer's law

vi) **Qualitative analysis:**

By comparing the absorption spectrum with the spectra of known compounds the quality of the newly synthesized compound can be studied .

vii) **Chemical kinetics:**

To study the kinetics of reaction,and in determination of dissociation constants of acids and bases from the change of absorption spectra with pH.

viii) **Determination of configurations of geometrical isomers:**

Cis-isomer suffers distortion and absorbs at lower wavelength as compared to trans-isomer

ix) **Detection of Functional Groups:**

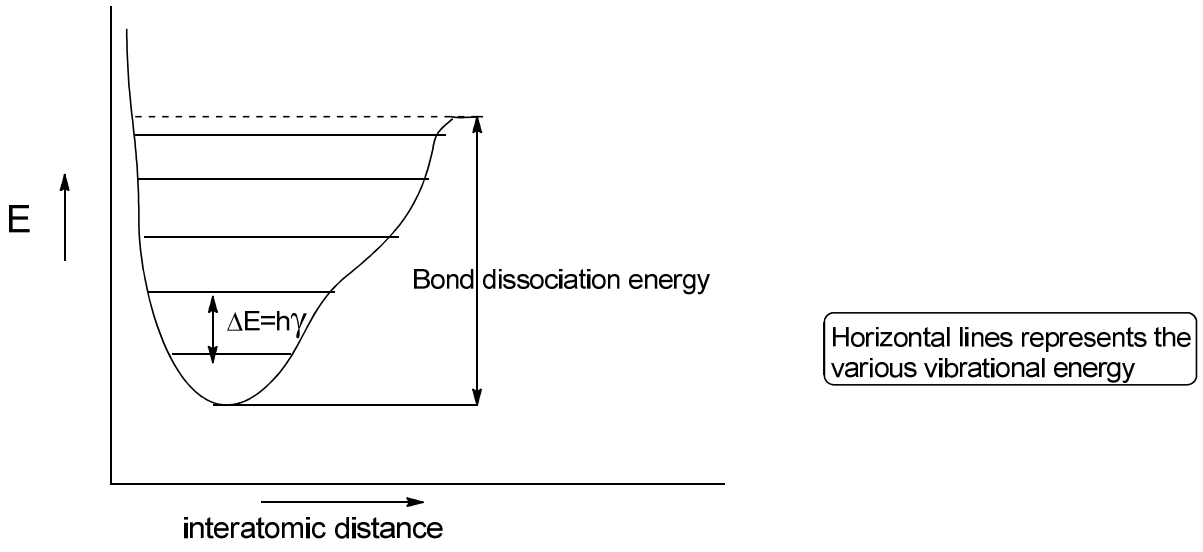
UV spectroscopy is used to detect the presence or absence of chromophore in the compound. Absence of a band at particular wavelength regarded as an evidence for absence of particular group

x) **In determining the structure of vitamins:**

It helps in predicting the relation between different groups this helps in determining the structure of several vitamins.

Infrared spectroscopy

Origins of Infrared spectrum (introduction)



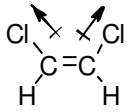
IR spectrum is considered as vibrational –rotational spectrum.IR radiation is not enough to produce the excitations of electrons; however, it causes vibrational excitation of the covalent bonds within that molecule. These vibrations are quantized and they occur only when the compound absorbs IR energy in a particular region.

The energy possessed by a molecule at any given moment is defined as the sum of the following contributing energy terms.

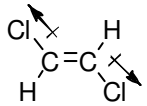
$$E_{Total} = E_{electronic} + E_{vibrational} + E_{rotational} + E_{Translation}$$

Absorption of appropriate electromagnetic radiation produces different excitations in the molecules. The fundamental requirement for absorption of an IR radiation is that there must be a net change in dipole momentum during the vibration for the molecule or the functional group under study. The change in the dipole moment of the molecule gives the absorption bands in the IR. Otherwise, if no absorption i.e. the net dipole moment is zero, they are said to be IR inactive. All the bonds in a molecule are not capable absorbing IR energy, but only those bonds which are accompanied by a change in dipole moment will absorb IR energy. Eg: CO and H-Cl absorb IR radiations, but the symmetrical molecules like H_2, N_2, and Cl_2 do not absorb IR radiation.

The below isomers show bands for C—H and C—Cl stretching, but only *cis*- form show C=C stretching.



μ = 2.95 D
cis-1,2-dichloroethane
show C=C stretching



μ = 0
trans-1,2-dichloroethane
no C=C stretching

Theory of Molecular vibrations (types of vibrational modes **<u>or</u>** fundamental frequencies**)**

The vibrational and the rotational energies of the molecules are increases with the passage of infra-red light through the sample. High energetic radiations change the molecular vibtaions and the low energy radiations change the molecular rotational levels.

The energy required to bring the changes in vibrational energies of a molecule depends on its

i) Molecular weight
ii) Bond strength
iii) The arrangement of atoms within the molecule.

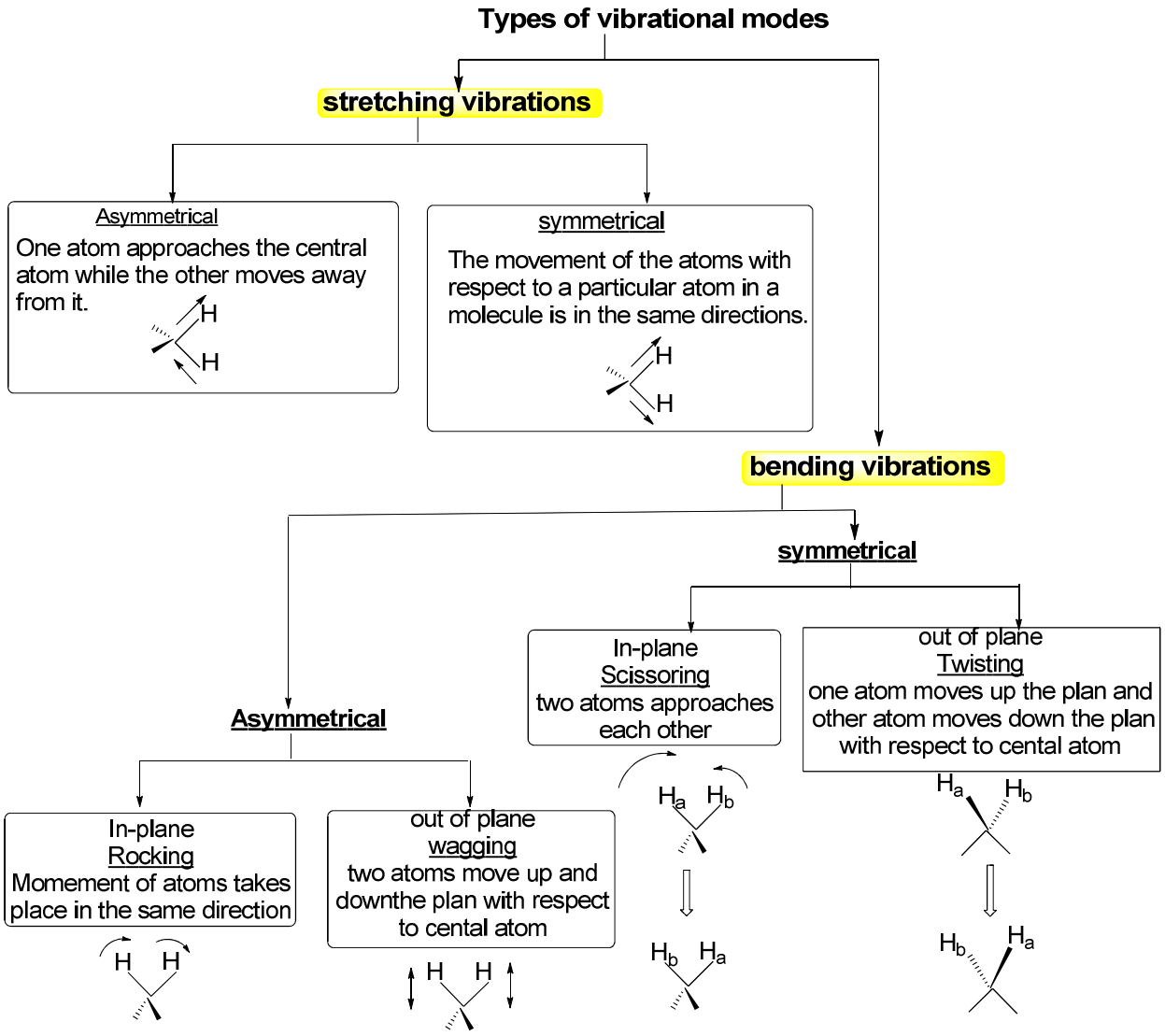
There are two types of molecular vibrations *1*) stretching vibrations *2*) bending vibrations.

1) <u>Stretching vibrations</u>: it is a rhythmical moment along the bond axis, such that the inter-atomic distance is increases or decreases but the atoms remains in the same bond axis.

2) <u>Bending vibrations</u>: It brings the change in bond angle between bonds and it does not occur along the axis of the bond. This leads to change in bond angle.

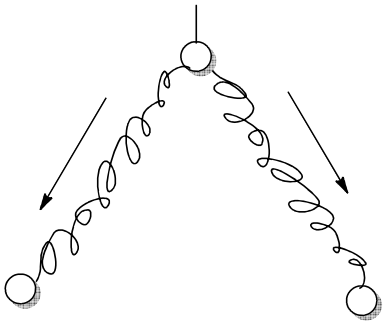
Energy required for Stretching vibrations > Bending vibrations. As the bending vibrations requires lesser energy and hence occur at higher wavelengths as compared to stretching absorption of the same band.

Each stretching and bending vibrations of a molecule occurs with a characteristic frequency. When a compound is exposed to an IR radiation of a frequency the molecules in it will absorb energy that exactly matches the frequency of one of its vibrations.

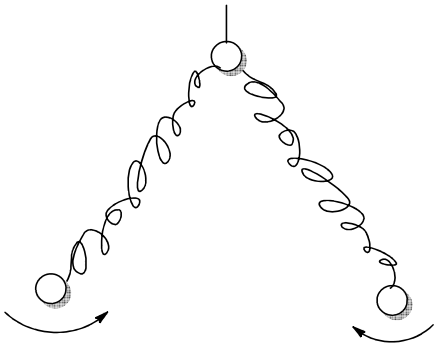


A diatomic molecule such as HCl and only undergo a stretching vibration.

The vibrations of a molecule containing three atoms are more complex. There are symmetric and asymmetric stretches and bends. Bending vibrations can be either in-plane or out of plane. Bending vibrations are often called as scissor, rock, twist and wag.



Stretching



Bending

IR Instrumentation (IR Spectrometer)

Principle of Infrared Spectroscopy:

The IR spectroscopy theory utilizes the concept that molecules tend to absorb specific frequencies of light that are characteristic of the corresponding structure of the molecules.

The instrumentation of infrared spectroscopy is illustrated below.

I

R radiation source:

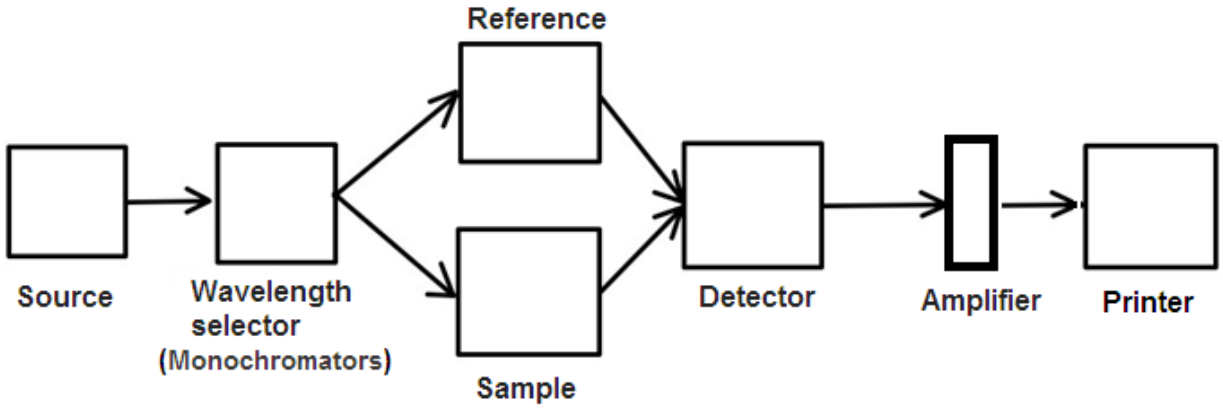
The Nernst Glower is electrically heated to about 2000 °C for the production of IR radiation. Nernst Glower consist of cylindrical hollow rod or tube having a diameter of 1- 2 mm and length of 30 mm. It is composed of a mixture of rare earth oxides such as zirconium oxide (ZrO_2), yttrium oxide (Y_2O_3) and thorium dioxide (ThO_2) sealed by platinum leads to the ends to permit electrical connection..

Monochromators:

A device used to select radiation of (or very close to) a single wavelength or energy. Sodium chloride is most commonly prism salt used

Sample:

IR spectroscopy has been used for the characterization of solids, liquids or gas samples. As a result a suitable sampling technique has to select. However, material containing sample must be transparent to the IR radiation.



Block diagram of typical IR spectrometer

Detector:

Detectors are used to measure the intensity of unabsorbed infrared radiation. Detectors like thermocouples, pyro-electric detectors and Photoconducting are used.

i) *Thermal detectors*- Their responses depend upon the heating effect of radiation

ii) *Pyroelectric detectors*- ability of certain materials to generate an electrical potential when they are heated or cooled.

iii) Photo conducting detectors- which are based on **photoconductive** semiconductor materials. The absorption of incident light creates non-equilibrium electrical carriers, and that reduces the electrical resistance across two electrodes.

Amplifier:

Helps to convert electrical signals into measurable physical quantities

Computer/Printer:

Computers are used to store the IR spectrum and for printing.

Working procedure of IR spectrometer:

First, a beam of IR light from the source is split into two and passed through the reference and the sample respectively. Now, both of these beams are reflected to pass through a detector. The data obtained from detector are converted into readable form with amplifier and finally the required reading is printed out.

In infrared spectrophotometer, IR radiation of successively decreasing frequency is passed through the sample of the compound and the percent (%) transmittance is measured. An IR spectrum is the graph of percent transmittance (%T) versus decreasing frequency expressed in wave numbers (4000—600 cm^{-1}).

APPLICATION OF IR SPECTROSCOPY

IR finds many applications in industry as well as in research because different molecules with different combination of atoms produce their unique spectra.

a) Identification of functional group and structure elucidation

The main advantage of infrared spectroscopy is the ability to confirm the presence of functional groups. It will give more accurate information about the presence of functional groups.

b) Identification of substances

If two compounds have identical IR spectra then both of them must be samples of the same substances. IR spectra of two enatiomeric compound are identical. So IR spectroscopy fails to distinguish between enantiomers.

c) Studying the progress of the reaction

Progress of chemical reaction can be determined by examining the small portion of the reaction mixture withdrawn from time to time. The rate of disappearance of a characteristic absorption band of the starting material and/or the rate of appearance of the characteristic absorption band of the product group due to formation of product is observed.

d) Detection of impurities

IR spectrum of the test sample to be determined is compared with the standard compound. If any additional peaks are observed in the IR spectrum, then it is due to impurities present in the compound.

e) Quantitative analysis

It is a dynamic measurement used for quantitative analysis.The quantity of the substance can be determined either in pure form or as a mixture of two or more compounds. This can be done by comparing the standard peak corresponding to the drug and test sample. This is called base line technique to determine the quantity of the substance

f) In forensic analysis

It is also employed in forensic analysis in civil and criminal analysis

What is difference between IR and FTIR?
IR stands for Infrared spectroscopy and **FTIR** stands for Fourier Transform Infraredspectroscopy.
IR spectroscopy gets absorption of a monochromatic **IR** light at a time and draws the spectrum, whereas,
FTIR, Multi-chromatic takes a summarized absorption of light and distributes it to create a spectrum using **FTIR**

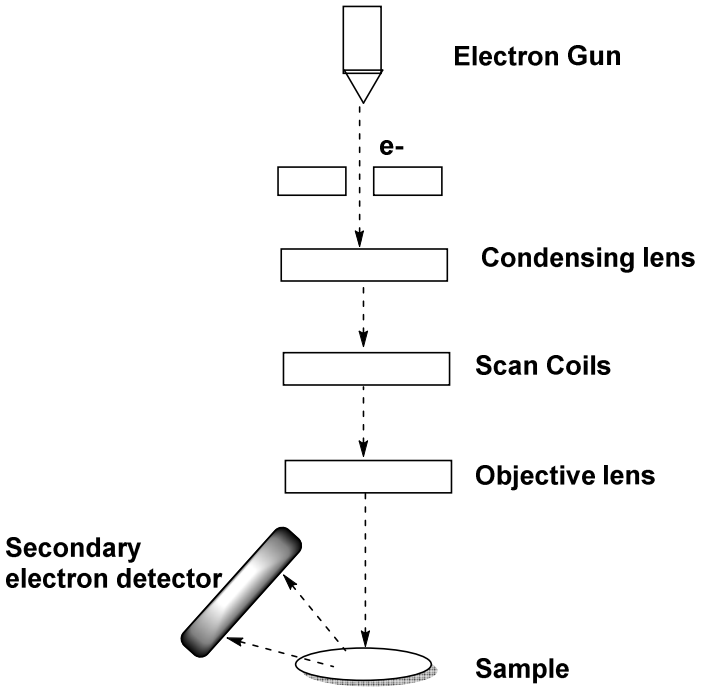
Principle and applications of physicochemical methods

Principle and applications of SEM

Principle: A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons.

The sample in SEM is exposed to the high-energy electron beam as a result electrons interact with atoms in the sample, producing various signals that gives information about topography, morphology, orientation of grains etc. of a material. Hence SEM is a very useful tool for the characterization of materials.

- Topography- the arrangement of atoms/ molecules on the surface of material
- Morphology- a particular form, shape, or structure appearing externally.
- crystallography - the arrangement of atoms in the materials



Block diagram of SEM

A schematic representation of an SEM is shown in Figure. Electrons are generated at the top of the column by the electron source and immediately they permit to pass through anode. Anode is a metal plate with a +ve charge, this attracts the electrons to form a beam. As result these primary electrons move more quickly down the column under vacuum to prevent the ionization of gases molecules. Scan coil helps to remove if any ions are formed.

Electromagnetic lenses are used to control the path of the electrons. Scanning coils are used to allow the beam onto the sample only.

When primary electrons are allowed to hit on the metal, results the secondary electron emitted from the surface of the sample. These two types of electrons used as signals in SEM imaging.

What are secondary electrons?

Secondary electrons are electrons generated as ionization products. They are called 'secondary' because they are generated by other radiation (the primary radiation). The primary radiation can be in the form of ions, electrons, or photons with sufficiently high energy, i.e. exceeding the ionization potential.

APPLICATIONS OF SEM

SEMs can be used in a variety of industrial, commercial, and research applications

a) Materials Science:

Used in investigation / analysis of nano materials (like nanotubes, nanofibres), superconductors, electronics etc.

b) Semiconductor Inspection:

Reliable performance of semiconductors requires accurate topographical information. SEM offers a speedy, accurate measurement of the composition of the semiconductor.

C) Microchip Assembly:

The high resolution, three-dimensional image of SEM is invaluable to design and production microchips.

d) Forensic Investigations:

Criminal and other forensic investigations utilize SEMs to uncover evidence and gain further forensic deep analysis. Uses include:

- analysis of gunshot residue
- bullet marking comparison
- handwriting and print analysis
- Examination of banknote authenticity.

e) Biological Sciences

SEM can be used on anything from insects and animal tissue to bacteria and viruses for study. Uses include:

- Measuring the effect of climate on change of species.
- identifying new bacteria and virulent strains
- vaccination testing
- in the field of genetics

f) Soil and Rock Sampling(Geological sampling)

SEM can determine weathering processes, to identify composition of rock.Valid uses include:

- Identification of tools and early human artefacts .
- soil quality measurement for farming and agriculture
- Dating historic ruins .
- Forensic evidence is soil quality, toxins etc.

g) Medical Science .

Broadly speaking, SEMs are used in medical science to compare blood and tissue samples in determining the cause of illness and measuring the effects of treatments on patients

Disadvantages of SEM

i) SEMs are expensive

ii) Need training to operate and for analysis of the sample.

iii) Must be placed in the area free of any possible electric, magnetic or vibration interference.

iv) The maintenance involves keeping a steady voltage, currents to electromagnetic coils

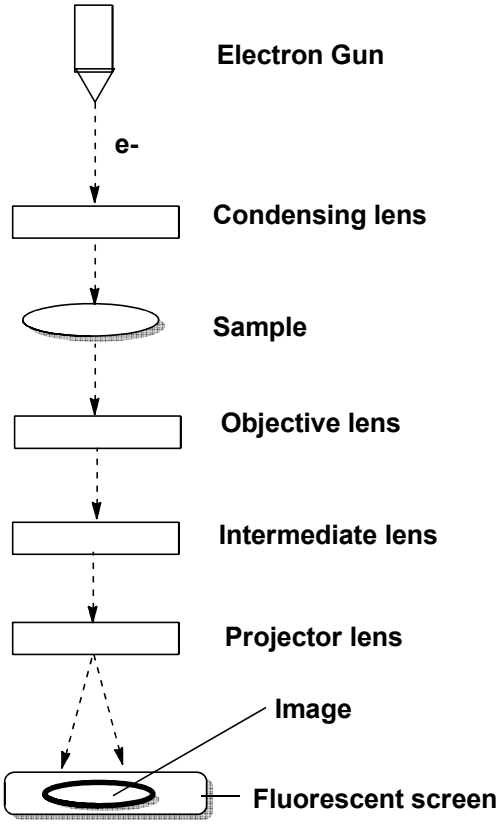
Principle and applications of TEM

A **Transmission Electron Microscope (TEM)** produces images via the interaction of electrons with a sample. They are also the most powerful microscopic tool available to-date for material science, capable of producing high-resolution, detailed images 1 nanometer in size.

Principle:

In TEM high energy beam of electrons is shone through a very thin sample and the interactions between the electrons and the atoms can be used to analyze the chemical sample.

Working:



Block diagram of TEM

A schematic representation of TEM is shown in Figure. A beam of high velocity electrons accelerated under vacuum (to avoid ionization/ interference of molecule in air) focused by condenser lens onto specimen and these electrons interacting with the specimen as it passes through. The emergent electron beam is focused by objective lens. Final image forms on a fluorescent screen for viewing. An image is formed from the interaction of electrons transmitted through the specimen. The image is magnified and focuses onto an imaging device, such as fluorescent screen or to be detected by a sensor such as CCD camera.

To obtain a TEM analysis, samples need to have certain properties:

i) The material need to be sliced thin enough for electrons to pass through it.

ii) Samples need to be able to withstand the vacuum chamber.

TEM Applications

TEMs have a wide-range of applications in a variety of scientific, education, research and industrial fields.

a) In structure analysis**:**

TEMs provide topographical, morphological, compositional and crystalline information.

i) The images allow researchers to view samples on a molecular level, making it possible to analyze structure and texture.

ii) This information is useful in the study of crystals and metals.

iii) TEMs provide information on element and compound structure

b) Industrial applications**:**

i) TEMs can be used in semiconductor analysis and production

ii) In the manufacturing and design of silicon chips.

iii) Technology companies use TEMs to identify flaws, fractures and damages to micro-sized objects; this data can help fix problems and/or help to make a more durable, efficient product.

c) In education and research**:**

i) Very useful in understanding the materials especially nano-sized

ii) Image morphology of chemical samples suspended on a thin film

d) In medical and pharmaceutical field**:**

i) For detailed studies of thin specimens (tissue sections, molecules, etc) for understanding the tissue arrangements and component.

ii) To study the small or whole organisms such as viruses or bacteria, and frozen solutions.

iii) Also in contaminant identification.

e) Forensic analysis**:**

TEM can be very useful in forensic analysis due to its ability to analyze from a very small particle.

f) Other fields**:**

To detect the internal structure of materials in gemology and metallurgy industry

Disadvantages of TEM

- TEMs are large and very expensive
- TEM sample preparation is a quite complex and tedious procedure
- Operation and analysis requires special training

- Samples are limited to those that are electron transparent, able to tolerate the vacuum chamber and small enough to fit in the chamber
- Images are black and white
- High maintenance cost. A Transmission Electron Microscope requires constant upkeep including maintaining voltage, currents to the electromagnetic coils and cooling water.

Which is better TEM or SEM?

- This all depends on what type of analysis want to perform. For example, SEM used to get information about the surface features of sample, like roughness or contamination detection. But the crystal structure of sample can be determined by using TEM only.
- SEMs provide a 3D image of the surface of the sample whereas TEM images are 2D image of a small area of the sample, which in some cases makes the interpretation of the results more difficult for the operator.

Difference between SEM and TEM

SEM	TEM
• A electron beam is focused to a fine point	• A broad static beam used
• SEM is based on scattered electrons	• TEM is based on transmitted electrons
• Scattered electrons in SEM produces the image of sample	• Electrons are directly pointed towards the sample
• SEM focuses on the sample's surface and its composition	• TEM seeks to see what is inside or beyond the surface
• SEM shows the sample bit by bit.	• TEM shows the sample as a whole
• SEM allows for large amount of sample to be analyzed at a time	• TEM only small amount of sample can be analyzed at a time
• SEM produces 3D images	• TEM gives 2D image
• SEM only offers 2 million as a maximum level of magnification	• TEM ha up to a 50 million magnification
• SEM has resolutions from 5 to 10 nm	• The resolution of TEM is 0 -1 nm
• No special effort required for sample preparation	• sample has to be cut thinner generally below 150 nm
• SEMs usually use acceleration voltages up to 30 kV	• TEM users can set it in the range of 60 – 300kV.

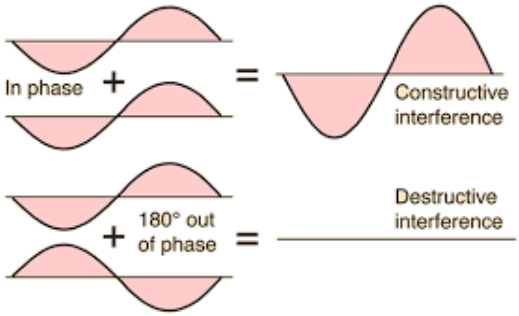
Principle and applications of X-ray diffraction

The principle behind XRD analysis**:**

XRD analysis is based on constructive interference of monochromatic X-rays and a crystalline sample. The interaction of the incident rays with the sample produces constructive interference

when conditions satisfy Bragg's law ($n\lambda = 2d \sin\theta$).

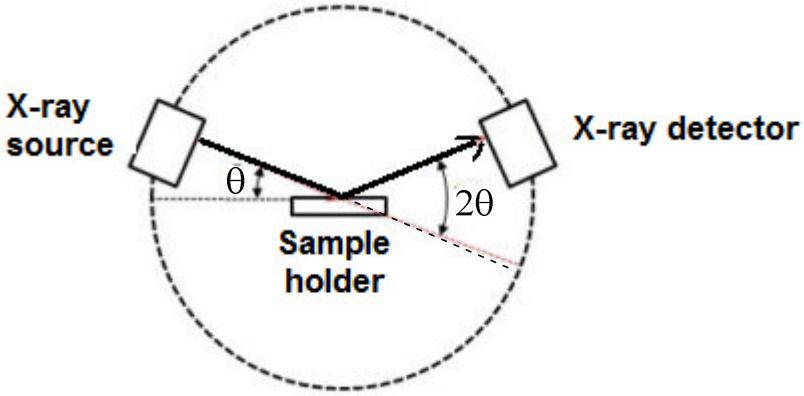
Constructive interference - when the maxima of two waves add together (the two waves are in phase), the amplitude of the resulting wave is equal to the sum of the individual amplitudes.



X-ray diffractometers consist of three basic units:

- X-ray tube also called as *cathode ray tube*
- Sample holder
- X-ray detector

- The X-rays are generated by **X-ray tube** (*cathode ray tube)*, filtered to produce monochromatic radiation, and directed toward the sample and the diffracted rays are collected.
- The key component of all diffraction is the angle between incident and diffracted rays.
- The sample rotates in the path of the collimated X-ray beam at an angle θ while the **X-ray detector** is mounted on an arm to collect the diffracted X-rays and rotates at an angle of 2θ.



Block diagram of XRD

Applications of XRD:

- XRD is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information of unit cell dimensions.
- For the identification of unknown crystalline materials (e.g. minerals, inorganic compounds).

- Determination of unknown solids is critical to studies in geology, environmental science, material science, engineering and biology.
- For measurement of sample purity
- XRD can be used to determine material structure and to determine the atomic arrangement
- To determine of modal amounts of minerals(quantitative analysis)
- For characterization of thin films samples. XRD studies helps to identify the chemical composition on the film and to determine the thickness, roughness and density of the film
- Forgiving final confirmation to the organic compound structures which are confirmed by various analytical techniques earlier.
- To know the exact grain size of the material that are difficult to determine optically.
- Measuring super lattices in multilayered epitaxial structures. .
- To make textural measurements, such as the orientation of grains in a polycrystalline sample.

Strengths of XRD

- Powerful and rapid (< 20 min) technique for identification of an unknown mineral
- Minimal sample preparation is required
- Data interpretation is relatively straight forward and simple

Limitations of XRD

- Homogeneous and single phase material is required for identification of structure of unknown compound
- Requires tenths of a gram of material which must be ground into a powder
- Peak overlay may occur and worsens for high angle 'reflections'

✓ This material gives you a basic information
✓ Refer text book for more details