CODEN [USA]: IAJPBB ISSN: 2349-7750

INDO AMERICAN JOURNAL OF
PHARMACEUTICAL SCIENCES
SJIF Impact Factor: 7.187
https://zenodo.org/records/10830664
https://www.iajps.com/volumes/volume11-february-2024/55-issue-02-february-24/

Available online at: http://www.iajps.com *Review Article*

A REVIEW ARTICLE ON THE INFRARED (IR) SPECTROSCOPY

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Abstract:

This review mainly focuses on the principal, instrumentation, interpretation, application of infrared spectroscopy. Infrared (IR) radiation refers broadly to that part of the electromagnetic spectrum between the visible and microwave region. Of greatest practical use to the organic chemist is the limited portion between 4000 and 400cm -1. Infrared spectroscopy monitors the interaction of functional groups in chemical molecules with infrared light resulting predictable vibrations that provides a "fingerprint" characteristic of chemical or biochemical substances present in the sample. Infrared spectroscopy is a technique that probes the vibrations within a material. Infrared spectroscopy has always been a powerful tool for the identification of organic materials. The development of Fourier transform infrared (FTIR) spectroscopy has introduced a popular method for the quantitative analysis of complex mixtures, as well as for the investigation of surface and interfacial phenomena.

Key words: *IR Spectroscopy, Detectors, Polyatomic molecules, Stretching Vibrations, Bending Vibrations.*

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Please cite this article in press ***Puduru Thogata Nagaraju*** *et al.,* ***A Review Article On The Infrared (IR) Spectroscopy****, Indo Am. J. P. Sci, 2024; 11 (02).*

INTRODUCTION:

The infrared (IR) spectroscopy deals with the study of absorption of infrared radiation by chemical compounds.
The IR radiation corresponds to the region between visible and microwave region of electromagnetic spectrum.
The IR spectrum is mainly used to determine the functional groups present in a compound and elucidate the structure of compound.

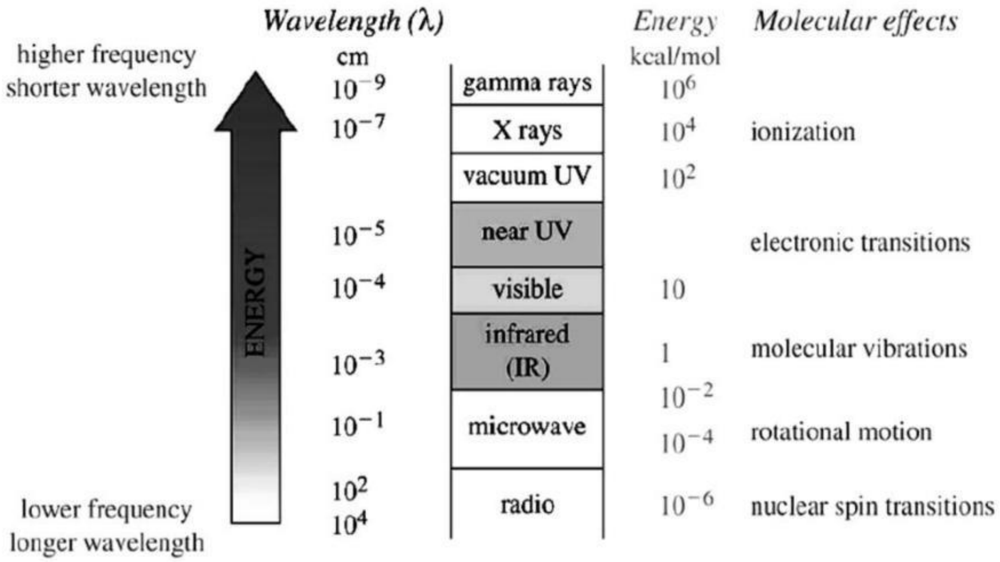

Fig.1. Various wavelength ranges of electromagnetic radiations (EMR)

Principle

The energy of a molecule=Electronic energy + Vibrational energy + Rotational energy

Different compounds when absorb infrared radiation give rise to vibrational transitions characteristic to the molecular structure and those transitions are recorded as IR Spectra.

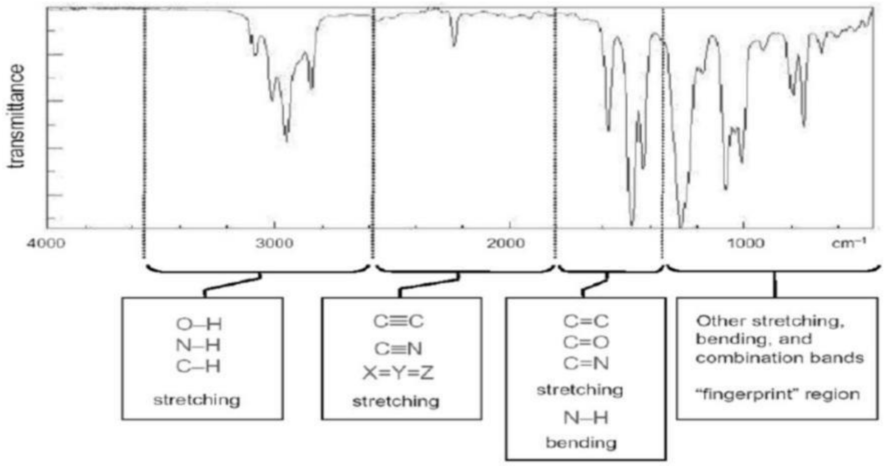

Fig.2. Typical IR spectrum depicting regions of absorption

The infrared range covers from 14,290—200 cm^{-1}. It is further subdivided into three regions:

1. Near IR region: 14290—4000cm $^{-1}$ (0.75 to 2.5μ)
2. Mid IR region: 4000—666cm $^{-1}$(25 to 2.5μ)
3. Far IR region:700— 200cm $^{-1}$(25 to 1000μ)

Out of these three regions the Mid IR region is widely used in pharmaceutical studies. Overall, the IR region is broadly divided into:

Finger print region (400-1500cm $^{-1}$): This region of IR spectrum provides characteristic peaks for different part of a molecule and helps in qualitative identification of compounds. Group frequency region (4000—1500cm $^{-1}$): Peaks with respect to different functional groups are observed in this region. Table 1 enlists various functional groups and their corresponding frequency regions.

Table 1. Characteristic IR frequencies of some known functional groups

Functional group	**Vibration Type**	**Freeequency(cm-1)**
ALCOHOL		
O-H	(stretch, H-bonded)	3200-3600
O-H	(stretch, free)	3500-3700
C-O	(stretch)	1050-1150
ALKANE		
C-H	stretch	2850-3000
-C-H	bending	1350-1480
ALKENE		
=C-H	stretch	3010-3100
=C-H	bending	675-1000
C=C	stretch	1620-1680
ALKYL HALIDE		
C-F	stretch	1000-1400
C-Cl	stretch	600-800
C-Br	stretch	500-600
C-I	stretch	500
ALKYNE		
C-H –	stretch	3300
	stretch	2100-2260

AMINE		
N-H	stretch	3300-3500
C-N	stretch	1080-1360
N-H	bending	1600
AROMATIC		
C-H	stretch	3000-3100
C=C	stretch	1400-1600
CARBONYL		
C=O	stretch	1670-1820
ETHER		
C-O	stretch	1000-1300
NITRILE		
C-N	stretch	2210-2260
N-O	stretch	1515-1560 & 1345-1385

Molecules consist of atoms or group of atoms that are connected by bonds which are analogous to springs. These bonds are flexible in nature in the molecules and they move continuously to gain vibrations having some frequency. This frequency of vibration is characteristic to the different parts of the molecule and hence, it can be called as the ***natural frequency of vibration.*** When we apply an external infrared frequency and it matches the natural frequency of vibration within a molecule the applied energy is then absorbed by the molecule and an infrared peak is observed. These peaks are characteristic to different functional group and parts of the molecule and they are unique for every molecule. Hence the IR spectroscopy is considered as the fingerprint analysis of a molecule. The IR region is measured in terms of Wave number and it is expressed as:

"It is the number of waves present per cm i.e. Wave number = 1/Wavelength (µm)

$\times\ 10^4$ A compound can be analyzed by IR radiation only if:

1. There is a change in dipole movement
2. There should be a resonance in applied and natural IR frequency

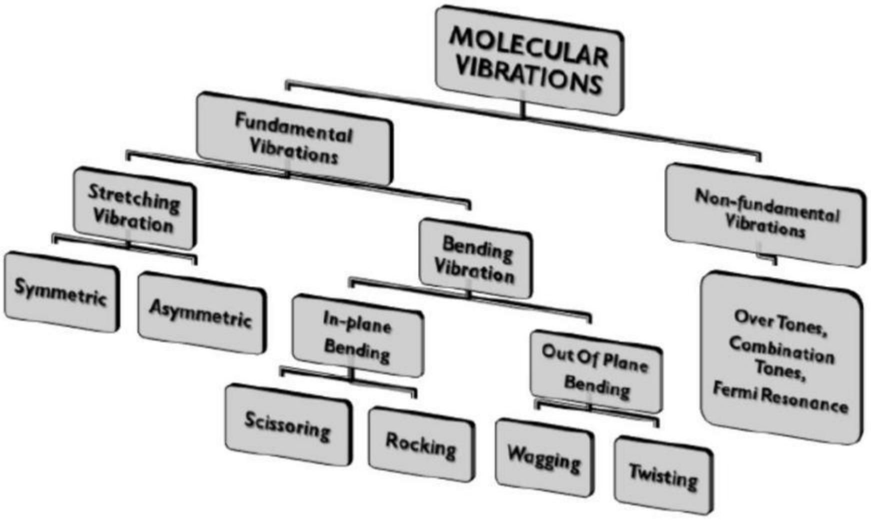

Fig. 3. Overall classification of molecular vibrations

Fundamental modes of vibrations in a polyatomic molecule
There are mainly two types of vibrations that are present within a molecule
They are:

1. Stretching vibration: In stretching vibration, the length of a bond is changed i.e. either increased or decreased. It is of two types:

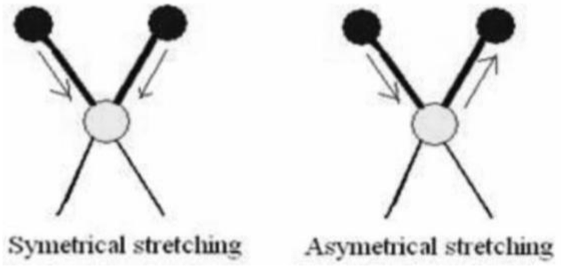

Fig.4. Types of stretching vibrations

A] Symmetrical stretching: Here the two bonds increase or decreases in length in a symmetric pattern.

B] Asymmetrical stretching: Here when one bond length increases the other ones decreases.

2. Bending vibration:

Bending vibrations are of two sub-types.

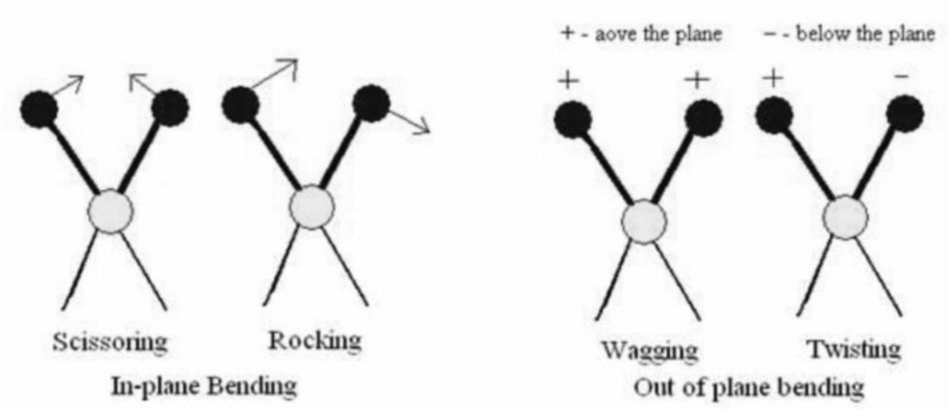

Fig. 5. Types of bending vibrations

A] In plane bending and we Out-of-plane bending:

When there is a change in bond angle during the vibration it is called is in plane bending and the bending of bonds occur within a plane. The implant bending can be again of two subtypes known as scissoring where the bond angle decreases and rocking with the bond angle is unaltered however both the bond moves within the same plane.

In case of out of plane bending the bending occurs in different planes of the molecule and is again subdivided into two types.

(a) Wagging where both atoms move to one side of the plane and
(b) Twisting where one atom remains above the plane whereas the other one below the plane.

The total number of fundamental vibrations for any given polyatomic molecule having "n” number of atoms can be determined using the expression:

A] 3n- 6 for the nonlinear molecule B] 3n- 5 for the linear molecule

However, few other kinds of vibrations can also be found in infrared spectrum known as overtones, combination bands, difference bands and Fermi resonance.

- The ***overtone*** vibrations occur at twice or thrice the value of fundamental vibrations.
- ***Combination bands*** are the bands that result because of coupling of fundamental frequencies

- ***Difference bands*** are noticed at the frequency which is actually the difference of two bands.

- ***Fermi resonance*** bands are produced because of coupling between fundamental vibration and an overtone or because of a combination band.

According to the expression stated above for linear or non-linear molecules it is very difficult to obtain the predicted number of vibrations. There may be a variation in the predicted number of bands and observed number of bands due to:
1] More number of bands may be produced due to:

- Overtones

- Combination bands

2] Less bands may be obtained:

- When a vibration doesn't involve a change in dipole moment

- When the fundamental bends fall outside the region of 4000 to 400 cm^{-1}
- When the bands are too weak to be observable

- When the vibrations occur very close to each other such that they coalesce

In a symmetrical molecule many absorptions with same frequency produce a degenerate band.

Polyatomic molecules have multiple atoms, and they can undergo various modes of vibration. The fundamental modes of vibration in polyatomic molecules can be categorized into three main types:
Stretching Vibrations:

Symmetric Stretching: In this mode, atoms move closer together or farther apart along the axis of the bond in a symmetrical manner. It involves the stretching or compression of the bond. For example, in CO2, the O-C-O bending.

Asymmetric Stretching:

In this mode, atoms on either side of the central atom move in opposite directions along the axis of the bond. It results in an asymmetric stretching of the bond. For example, in CO2, the O=C=O stretching.

Bending Vibrations:

Scissoring: In this mode, two groups of atoms move in opposite directions, like a pair of scissors closing and opening.

Rocking: In this mode, a group of atoms moves in one direction, while another group moves in the opposite direction, causing a rocking motion.

Twisting/Torsional: In this mode, groups of atoms rotate with respect to each other around a central bond. This usually involves dihedral angle changes.

Dilational (Volume) Vibrations: In this mode, the entire molecule expands or contracts uniformly without any change in bond angles or the overall shape.

Polyatomic molecules can exhibit various combinations of these fundamental modes, leading to complex vibrational spectra in their IR (Infrared) spectra. The number and types of vibrational modes depend on the molecule's geometry and the number of atoms and bonds it contains. The study of these vibrational modes in IR spectroscopy is essential for characterizing and identifying polyatomic molecules.

Sample handling

The sample cells are usually made up of transparent ionic substances such as sodium chloride

(NaCl) or potassium bromide (KBr). However, KBr is preferred because of its compatibility with the instrumental measurement process. The infrared samples can be considered of three forms such as:

Liquids:

- A drop of liquid is placed within the KBr plates with the thickness less than 0.01 mm.
- Around 1-10mg of sample is required to prepare the solution.
- Aqueous solutions are not preferred as they tend to dissolve the sample cells so the solvent should be anhydrous and mostly organic solvents such as chloroform is preferred.

Solid solutions:

- To prepare a solid solution 1 to 10 mg volumes of 0.1 to 1.0 ml of 0.05 to 10% solution are required for placement within a cell of 0.1 to 1mm thickness.
- CCl4 is used for this purpose which absorbs strongly at 785 cm^{-1}.
- Care should be taken to avoid solution combinations that react instantaneously.

Solids:

- The solid samples can be prepared using pressed pellet technique where the sample is placed in KBr pellets are prepared in form of thin transparent layer using a hydraulic press.
- However, as KBr is hygroscopic in nature care should be taken to minimise its atmospheric exposure.
- Another technique called as Nuzol-Mull technique (popularly called as Mull Technique) may be used where the mixing up of the sample with a mineral oil (Nujol) is done and afterwards a thin film of the liquid can be applied on the liquid sample cell for measurements.

Factors effecting vibrations:

Following are the factors that affect vibrational frequencies in a molecule **A]**

Coupled vibration

- An isolated C-H bond has only one stretching vibration frequency where as methylene group shows two stretching vibrations both symmetrical and asymmetrical.
- Because of mechanical coupling or interaction between CH stretching vibrations in the CH2 group.
- Asymmetric vibrations develop at higher frequency or wave numbers than stretching vibrations.
- Such vibrations are called coupled vibrations because these vibrations occur at different frequency then the required for an isolated CH stretching.

Fermi resonance

- Coupling of two fundamental vibration modes produces 2 new modes of vibration.
- With frequencies higher and lower than the observed in absence of interaction.

- This interaction takes place between fundamental vibrations and overtones or combination tone vibration and they are collectively called us Fermi resonance.

- In Fermi resonance molecule transfer energy from fundamental vibrations into overtone or combination level and back.

- The resonance pushes the two levels apart and mixes their character and each level has some amount of fundamental and some amount of overtone or combination tone characters.

B] Electronic effects

- Changing the absorption frequencies for a particular group takes place when the substituent in the neighbourhood of the particular group is changed.

- They include

    - Inductive effect: Inclusion of alkyl group gives +ve Inductive effect producing decreased absorption frequency. Adding an electronegative atom or group produces –ve inductive effect leading to increased absorption frequency.

    - Mesomeric effect: It produces lengthening or weakening of a bond leading to lowering of absorption frequency.

    - Field effect: In ortho substituted compounds, the lone pair of electrons on two atoms influence each other through space interactions and change the vibrational frequencies of both the groups. Example: Ortho halo acetophenone. This is called field effect.

C] Hydrogen bonding

- It occurs in any system having a proton donor group and a proton acceptor. If theS- orbital of the proton effectively overlaps the P or orbital of the acceptor group.

- The stronger the hydrogen bond, the longer the OH bond, the lower the vibration frequency and broader and more intense will be the absorption.

- Intermolecular bonds produce broad bands whereas intramolecular hydrogen bonds produce sharp and well defined bands.

Instrumentation

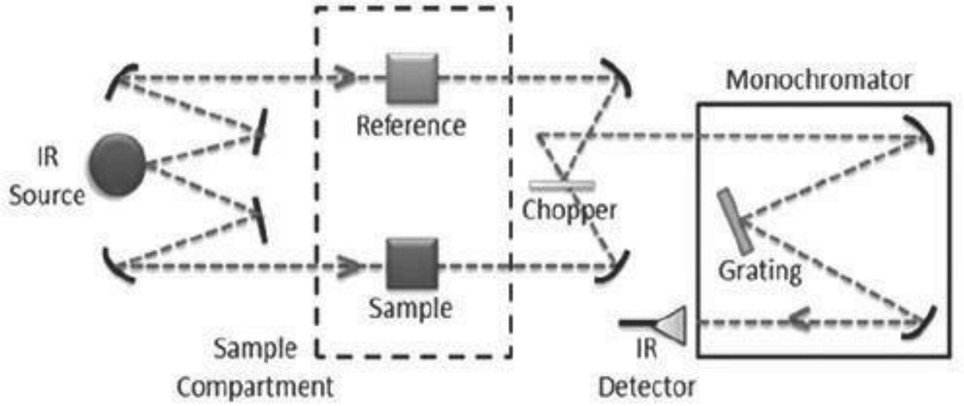

Fig. 6. Schematic representation of IR spectrometer

Sources of radiation

- The source of energy should produce very narrow beam of IR radiation
- The radiation should be intense enough to detect the analyte
- It should be steady
- It should cover the desired wavelength range

Following are some of the sources of IR radiation used in IR spectrophotometer:

1.Incandescent lamp

- This lamp is particularly used in near IR instruments. However, it is least preferred over other sources as it has a low spectral emission.

2. Nernst Glower

- It contains a hollow rod composed of rare earth oxides such as is it zirconia, yttria and thoria.
- It is in non-conducting at room temperature and requires heating by external means to bring it to a conducting state.
- The glower is heated to a temperature within the range of 1000-1800° C.
- It gives a maximum radiation of 7100 cm^{-1}

3.Globar source

- It is a rod prepared from centred Silicon carbide
- it is heated up to a temperature between 1300-1700°C
- it emits maximum radiation at 5200cm^{-1}
- It has a disadvantage that its radiation is less intense then Nernst glower

4. Mercury arc lamp

- It is a device made up of quartz-jacketed tube containing mercury vapour inside it at a pressure greater than 1 atmosphere.
- It is highly effective in the far-IR region where the other sources of radiation fail to provide continuous radiation.

Wavelength selectors

- They help in selecting a continuous IR radiation in the desired wavelength region.
- They generally contain a chopper and a complex system of monochromators.
- The choppers are generally used to enhance the signal to noise ratio of the instrument and they also moderate the intensity of radiation that reaches to the detector.
- The monochromator is used to select it desired frequency of radiation which will be then allowed to incident on the detector.

- These monochromators are of two types:

1. Prismatic monochromator: They are made up of glass/quartz and coated with alkyl halides.

A] Mono Pass (Radiation passes only once through the prism) B] Double Pass (Radiation passes twice through the prism)

2. Grating monochromator: They are grooves made up of aluminium and provide better dispersion of radiation than prisms.

A]Reflection gratings
B]Transmittance grating

Detectors:

1. Golay cell:

- It contains a small metal cylinder which is closed which is closed by black metal plate at one end and buy a flexible metal die programme at the other end

- The cylinder is filled with xenon and sealed.

- When IR radiation is allowed to fall on the black metal plate it heats the gas which expands it.

- The signal detected by the photo tube is then modulated according to the power of radiant beam.

2. Bolometer:

- It is made up of a thin metal conductor.

- When radiation falls on this conductor its temperature changes as the resistance of a metallic conductor changes with temperature the degree of change in resistance is regarded as the measurer amount of radiation that is incident on the bolometer.

- It follows the principle of Wheatstone bridge.

- When there is no radiation incident on the bolometer the bridge remains balanced and in case a radiation is incident on the bolometer the bridge becomes unbalanced due to the changes in electrical resistance and there is a flow of current through the galvano meter.

3. Thermocouple:

- It is based on the principle that electric current flows when there are two dissimilar metal wires connected together at both the ends and a temperature difference is present between the two ends.

- The end exposed to the IR spectrometer is called the Hot junction.

- To increase the energy gathering efficiency it is usually made up of black body.

- The other connection i.e. Cold junction is thermally insulated and carefully protected from stray light.

- The electricity flowing through is directly proportional to the energy difference between the two connections.

4. Thermistor:

- Thermistors are usually made up of fused mixture of metallic oxides.

- As the temperature of the mixture increases its electrical resistance decreases and accordingly the measurements are performed in the higher spectrophotometer.

5. Pyroelectric detector:

- The pyroelectric detector is a thermal sensor of infra-red radiation requiring no bias.
- While in principle a pure capacitor (hence theoretically noiseless), the detector has a varying noise contribution as a function of frequency due to a load resistor, series loss resistance, and amplifier.
- The actual sensor is a pyroelectric crystal exhibiting spontaneous polarization.
- The spontaneous polarization and dielectric constant of the crystal are temperature-dependent.
- A change in incident power raises the detector temperature causing an electric charge to appear across the electrode surfaces cut perpendicular to the crystal's ferroelectric axis.
- The evacuated detector package incorporates an electroded flake of triglycine sulfate mounted on a substrate of low thermal and electrical conductivity, a field effect transistor, load resistor, and an infrared transparent window.

Applications:

IR spectroscopy is applied for qualitative as well as quantitative analysis of drugs in Pharmaceutical industry.

- It is used for identification of drug substances.
- It identifies the impurities present in a drug sample.
- It helps in study of hydrogen bonding both intermolecular and intramolecular type.
- It is widely used in study of polymers.
- It helps determining issue of *Cis*- *Trans* isomers present in a mixture of compounds.
- It elucidates reaction mechanisms.
- It is a great tool investigation of rotational isomerism.
- It identifies functional groups present in any sample.
- It estimates relative stability of confirmation.
- It distinguishes between Sis and trance isomers.
- It can predict the keto-enol tautomerism.
- It helps in establishing quality of tea leaves.
- Identification of functional groups & structure elucidation of organic compounds.
- Quantitative analysis of a number of organic compounds.

- Study of covalent bonds in molecules.
- Studying the progress of reactions.
- Detection of impurities in a compound.
- Ratio of cis-trans isomers in a mixture of compounds.
- Shape of symmetry of an inorganic molecule.
- Study the presence of water in a sample.
- Measurement of paints and varnishes.

Infrared (IR) spectroscopy has a wide range of applications in various fields due to its ability to provide information about the chemical composition, molecular structure, and functional groups of substances. Some common applications of IR spectroscopy include:

- **Chemical Analysis**: IR spectroscopy is widely used in chemistry for identifying and characterizing organic and inorganic compounds. It can determine the presence of specific functional groups in a molecule, aiding in compound identification.
- **Pharmaceuticals**: In the pharmaceutical industry, IR spectroscopy is used for quality control and validation of drug formulations. It can detect impurities, analyze drug content, and assess the stability of pharmaceutical products.
- **Environmental Monitoring**: IR spectroscopy is used to analyze air and water samples to detect pollutants, such as volatile organic compounds (VOCs) and gases. It helps assess environmental quality and compliance with regulations.
- **Food and Beverage Analysis**: IR spectroscopy is employed for food safety and quality control. It can identify the composition of food products, detect contaminants, and assess nutritional content.
- **Polymer Characterization**: IR spectroscopy is crucial in the polymer industry for analyzing the chemical structure and composition of polymers. It helps in quality control and material development.

1. **Petroleum and Petrochemicals**: It is used to analyze crude oil, refined petroleum products, and petrochemicals. IR spectroscopy can identify hydrocarbons, determine oil properties, and assess product quality.

- **Forensic Science**: IR spectroscopy is used to analyze forensic evidence, such as drugs, explosives, and unknown substances found at crime scenes. It aids in identifying and characterizing evidence.
- **Art and Cultural Heritage Conservation**: IR spectroscopy is used to study artwork and historical artifacts to identify pigments, coatings, and the condition of the materials, aiding in preservation and restoration efforts.
- **Medicine and Healthcare**: IR spectroscopy is used for biomedical research and diagnostics. It can identify biomolecules, study cell and tissue samples, and detect diseases.
- **Material Science**: In material science, IR spectroscopy helps characterize materials like ceramics, semiconductors, and superconductors. It assesses material properties and structural changes.
- **Geology and Mineralogy**: It is used for mineral identification, especially in the study of rocks and minerals. IR spectroscopy can determine mineral composition and mineralogical information.

- **Textile Industry**: IR spectroscopy assists in the analysis and quality control of textiles and fibers, helping to assess fiber composition and properties.
- **Homeland Security**: IR spectroscopy is used for security purposes to detect hazardous materials, explosives, and chemical threats.

- **Research and Development**: It plays a critical role in scientific research across various disciplines, allowing researchers to gain insights into the molecular structures of substances.

These are just a few examples of the wide-ranging applications of IR spectroscopy. Its versatility and non-destructive nature make it a valuable tool in various industries and research fields for chemical analysis and material characterization.

Sample cells & Sampling techniques:

Gases

- Gas cell – 10cms

- Multi pass gas cells

Liquids

Thin film squeezed between 2 IR transparent windows.

□ 0.1 - 0.3mm thickness mm thickness.

Below 250cm-1 _ CsI flats, sample with water CaF2 flats □ Solids.

Four techniques:

1. KBr discs/ pellets/ pressed pellet technique

2. Mulls

3. Deposited films

4. Solutions

1.KBr discs:

- 0.1 – 2.0% by wt.

- Particle size - □ 2□m.

- Hydraulic pressure – 10 tons load.

- Discs: 13mm- diameter, 0.3mm- thickness.

2.Mulls:

- Grinding sample with a drop of oil.

- Nujol (liquid paraffin)

- Complement: Hexacholorobutadiene & chlorofluorocarbon.

3.Deposited films:

- Solution in a volatile solvent on a NaCl flat.

4.Solutions:

- Solvent – CCl4 , CS2 , CHCl3

- Complementary pair - CCl4 & CS2

Detectors
two main types in common IR instruments

a) Thermal Detectors
1.) Thermocouple - two pieces of dissimilar metals fused together at the ends

- when heated, metals heat at different rates

- potential difference is created between two metals that varies with their difference in temperature

- usually made with blackened surface (to improve heat absorption)

- placed in evacuated tube with window transparent to IR (not glass or quartz)

- IR “hits” and heats one of the two wires.

- can use several thermocouples to increase sensitivity.

2.) Bolometer
- strips of metal (Pt, Ni) or semiconductor that has a large change in resistance to current with temperature.

- as light is absorbed by blackened surface, resistance increases and current decreases

- very sensitive.

b) Photo conducting Detectors
- thin film of semiconductor (ex. PbS) on a nonconducting glass surface and sealed in a vacuum.

- absorption of light by semiconductor moves from non-conducting to conducting state.

- decrease in resistance □ increase in current.

- range: 10,000 -333 cm-1 at room temperature.

c) Pyroelectric Detectors
- pyroelectric (ceramic, lithium tantalate) material get polarized (separation of (+) and (-) charges) in presence of electric field.

- temperature dependent polarization.

- measure degree of polarization related to temperature of crystal - fast response, good for

FTIR

Fourier Transfer IR (FTIR) – alternative to Normal IR.

- Based on Michelson Interferometer

1) light from source is split by central mirror into 2 beams of equal intensity.

2) beams go to two other mirrors, reflected by central mirror, recombine and pass through sample to detector.

3) two side mirrors. One fixed and other movable.

a) move second mirror, light in two-paths travel different distances before recombined.

b) constructive & destructive interference.

c) as mirror is moved, get a change in signal.

Advantages of FT IR Spectrophotometer

FT-IR spectrometers have numerous performance advantages over traditional dispersive infrared instrumentation. Virtually all infrared spectrometer manufacturers are now using FT designs designs instead instead of dispersive dispersive. The benefits of upgrading to an FT-IR from an existing dispersive infrared instrument will be immediately evident in spectral quality, data collection speed, reproducibility of data, and ease of maintenance and use.

IR Spectrophotometer: Advantages

1. All kinds of material can be analyzed.

2. Provides lot of information.

3. Fast and easy.

4. Very small amount of sample is required.

5. Less expensive.

Disadvantages

1. IR spectra cannot be obtained for Mono atomic substances e.g.: helium, argon.
2. IR spectra cannot be obtained for Homonuclear diatomic molecules like O2 , N2 .
3. Works best for pure substances but not for complex mixtures.

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 Regions

In general, the IR spectrum can be split into four regions for interpretation:

4000 □ 2500 cm-1: Absorption of single bonds formed by hydrogen and other elements e.g. O□H, N□H,

C□HIR Absorption Regions 53

2500 □ 2000 cm-1: Absorption of triple bonds e.g. C≡C, C≡N

2000 □ 1500 cm-1: Absorption of double bonds e.g. C=C, C=O

1500 □ 400 cm-1: This region often consists of many different, complicated bands, called the fingerprint region. It is rarely used for identification of particular functional groups.

LIMITATIONS OF IR SPECTROSCOPY

*Cannot determine the molecular weight of the compound.4v2t
*Does not give information about the relative position of different functional groups in a molecule.
*From the single IR spectrum of an unknown substance,it is not possible to know whether it is pure compound or a mixture of compound.
*Sample cells are made of halogen salts which are susceptible to moisture.
*Gas samples cannot be analysed as they lack sensitivity.

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