Chapter
24

Analytical Chemistry

Major Concepts

24.1 Classical Method of Analysis
24.2 Modern Methods

Learning Outcomes:

Students will be able to:

- Compare the classical method of analysis with modern methods. **(Understanding)**
- Discuss the procedure of combustion analysis. **(Understanding)**
- Define spectroscopy and discuss its applications in analytical Chemistry. **(Applying)**
- State the regions of electromagnetic spectrum used in IR and UV/Vis spectroscopy. **(Applying)**
- Explain the origin of IR absorption of simple molecules. **(Analyzing)**
- Predict whether a given molecule will absorb in the UV/Vis region. **(Analyzing)**
- Predict the colour of a transition metal complex from its UV/Vis spectrum. **(Analyzing)**
- Outline in simple terms the principles of proton NMR spectroscopy. **(Applying)**
- Explain how chemical environment of a Proton affects the magnetic field it experiences and hence the absorption of energy at resonance frequency. **(Applying)**
- Describe standard scales used in proton NMR. **(Applying)**
- Explain instrumentation and working of MS. **(Applying)**
- Outline the use of MS in determination of relative isotopic masses and isotopic abundance. **(Applying)**
- Define and explain atomic emission and atomic absorption spectrum. **(Understanding)**

Introduction

Analytical chemistry can be considered as one of the essential branches of chemistry. It can be defined as the branch of chemistry in which we study about the separation, identification and quantification of components of samples of matter. Analytical chemist uses instruments and methods to separate, identify, and quantify matter. Analytical chemistry plays an important role not only in the fields of chemistry such as organic, inorganic, physical, nuclear and environmental, but also in the other fields of science such as physics, biology, archaeology, agriculture and industries. Because all these fields require analysis of materials.

Classification of Chemical Analysis

The process of estimation or determination of the components present in given sample is known chemical analysis. Chemical analysis procedures are usually classified in two ways:

i) In terms of the goal of analysis.

ii) In terms of the nature of analysis.

Classification in terms of Goal of Analysis

The chemical analysis can be classified into qualitative and quantitative analyses in terms of the goal of analysis.

Qualitative analysis

Qualitative analysis means what constituents are present in the sample. It is the determination of the chemical composition of a sample of matter. It can tell you whether an atom, ion, functional group, or compound is present or absent in a sample, but it does not provide information about its quantity.

There are two main types of qualitative analysis: organic compounds analysis and inorganic compounds analysis.

Organic compounds analysis involves the determination of atoms or functional groups in the sample. Examples include: (i) combustion analysis (ii) detection of elements in an organic compound (iii) identification of functional groups in an organic compound and (iv) identification of organic pollutants in the air.

Inorganic compounds analysis finds elemental composition of inorganic compounds. It is used to identify and separate cations and anions in a sample. Example for inorganic compounds analysis is salt analysis. The common qualitative test, used in inorganic chemical analysis, is the flame test. Flame tests can be used to identify the presence of a particular metal. Different metals give different colours to the flame.

Quantitative Analysis

Quantitative analysis means how much constituents is present in the sample. It is carried out when the identity of the components already known and when it is necessary to also know the amounts of these components. It is the determination of the amounts or concentrations of one or more components present in a sample. It is done by either a classical or modern (instrumental) procedure. For quantitative analyses, the amount of analyte (the material being analyzed) is determined by gravimetric or by titrimetric measurement.

In **gravimetric analysis**, the substance being determined is converted into an insoluble precipitate which is collected and weighed.

In **titrimetric analysis** (also known as volumetric analysis) the substance to be determined is allowed to react with suitable reagent added as a standard solution and the volume of solution needed for whole reaction is determined. The common titrimetric reactions are acid-base reactions, precipitation reactions and oxidation-reduction reactions.

Classification in terms of the Method of Analysis Used

The chemical analysis can also be classified into classical and instrumental methods in terms of the nature of method used.

24.1 Classical Method of Analysis

Classical methods are also known as wet chemical methods or earliest methods of chemical analysis. They are the traditional methods of chemical analysis which are still being used by chemists. The classical methods that were used to separate the components of interest in a sample were distillation, crystallization, extraction or precipitation. These methods usually involve the chemical reactions and/or classical reaction stoichiometry, but no electronic instruments are used other than an analytical balance or volumetric apparatuses such as pipette, burette etc. The instruments of classical methods are not highly specialized.

Classical methods are cheaper and easily available for labs of schools, colleges and industries. The classical methods consume more time than instrumental methods of analysis. Classical methods are more useful for accurate and precise measurements of analyte concentrations (at the 0.1% level or higher).

The Combustion Analysis

Combustion analysis is one the most widely used method for determining percentage composition and empirical formula of an unknown compound in the laboratory. This method is commonly used for compounds containing carbon and hydrogen.

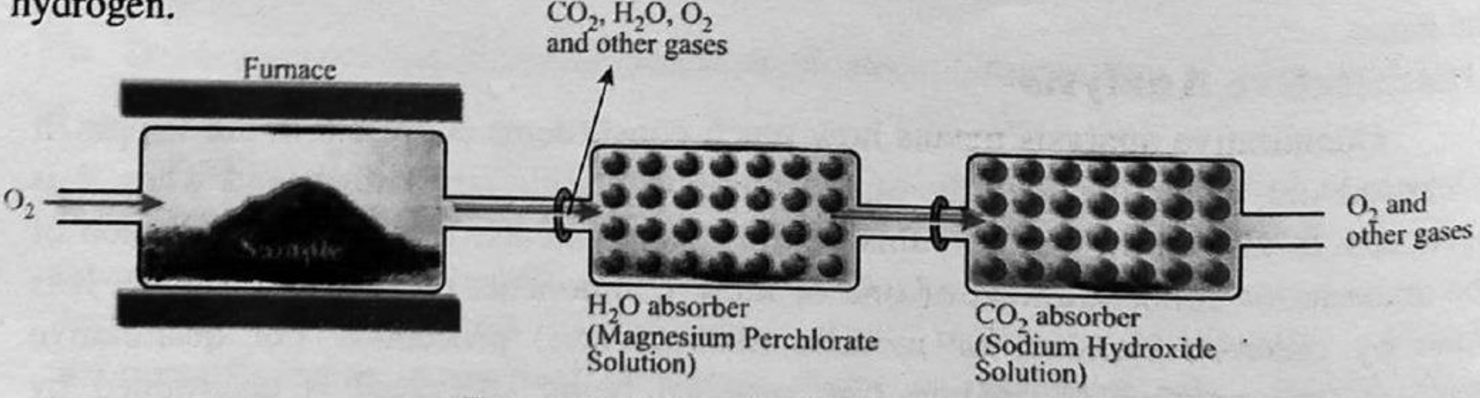


Figure: 24.1: Combustion Apparatus

In this method, a known mass of compound is burnt in stream of oxygen gas. Here each element in the compound combines with oxygen to produce volatile combustion products (CO_2 and H_2O). The carbon is converted to carbon dioxide and the hydrogen is converted to water vapours. Water vapours are absorbed in magnesium perchlorate, $Mg(ClO_4)_2$ whereas carbon dioxide is absorbed in 50% potassium hydroxide, KOH solution. The masses of CO_2 and H_2O are determined by measuring the mass increase in the CO_2 and H_2O absorbers. From the masses of CO_2 and H_2O we can calculate the percentage of carbon and hydrogen with the help of following equations:

$$\%\text{ age of carbon in compound} = \text{Mass of } CO_2 \times \frac{12}{44} \times \frac{100}{\text{Mass of compound}}$$

$$\%\text{ age of hydrogen in compound} = \text{Mass of } H_2O \times \frac{2}{18} \times \frac{100}{\text{Mass of compound}}$$

If third element such as oxygen is present in the compound, its percentage can be calculated by subtracting the percentages of carbon and hydrogen as:

$$\%\text{ age of oxygen in compound} = 100 - (\%\text{ age of carbon} + \%\text{ age of hydrogen})$$

Combustion analysis can be used to determine the empirical formula of an unknown organic compound.

Example 24.1:

Combustion of 1.125 g of an organic compound produces 3.447g of CO_2 and 1.647 g of H_2O. Calculate the % age of carbon and hydrogen.

Data:

Mass of organic compound = 1.125g
Mass of CO_2 = 3.447g
Mass of H_2O = 1.647g
%age of carbon = ?
%age of hydrogen = ?

Solution:

% age of carbon in organic compound

$$= \text{Mass of } CO_2 \times \frac{12}{44} \times \frac{100}{\text{Mass of compound}}$$

$$= 3.447\text{g} \times \frac{12}{44} \times \frac{100}{1.125\text{g}}$$

$$= 83.6\ \%$$

% age of hydrogen in organic compound

$$= \text{Mass of } H_2O \times \frac{2}{18} \times \frac{100}{\text{Mass of compound}}$$

$$= 1.647\text{g} \times \frac{2}{18} \times \frac{100}{1.125\text{g}}$$

$$= 16.3\ \%$$

Determination of Empirical Formula

The following steps are used to determine the empirical formula:

i) Divide the % by the atomic mass of each element to calculate the mole ratios.

$$\text{Mole ratio} = \frac{\%\text{age of an element}}{\text{Atomic mass}}$$

ii) Determine simple atomic ratio by dividing number of mole ratio of each element by the smallest one.

iii) If atomic ratios are in fractions, change them into whole numbers by multiplying with a suitable number.

iv) Write atomic ratios as subscript with each element to get empirical formula.

Example 24.2:

Caffeine, central nervous system stimulant, is found is tea and coffee, and has 49.48% carbon, 5.19% hydrogen, 16.48% oxygen and 28.85% nitrogen. Calculate its empirical formula.

Data:

%age of carbon = 49.48%
%age of hydrogen = 5.19%
%age of oxygen = 16.48%
%age of nitrogen = 28.85%
Empirical formula of caffeine = ?

Solution:

i) Calculate mole ratio of each element.

$$\text{Mole ratio of carbon} = \frac{49.48\%}{12.01\,\text{amu}} = 4$$

$$\text{Mole ratio of hydrogen} = \frac{5.19\%}{1.01\,\text{amu}} = 5$$

$$\text{Mole ratio of oxygen} = \frac{16.48\%}{16.00\,\text{amu}} = 1$$

$$\text{Mole ratio of nitrogen} = \frac{28.85\%}{14.01\,\text{amu}} = 2$$

ii) Determine simple atomic ratio

$$\text{Atomic ratioof carbon} = \frac{4}{1} = 4$$

$$\text{Atomic ratio of hydrogen} = \frac{5}{1} = 5$$

$$\text{Atomic ratio of oxygen} = \frac{1}{1} = 1$$

Atomic ratio of nitrogen $= \frac{2}{1} = 2$

iii) The empirical formula of caffeine is $C_4H_5ON_2$

Determination of Molecular Formula

Following steps are used to calculate molecular formula:

i) Divide the molecular formula mass by empirical formula mass to get value of n.

$$\text{Value of } n = \frac{\text{Molecular Formula Mass}}{\text{Empirical Formula Mass}}$$

ii) Multiply number of atoms of each element with value of 'n' to get molecular formula.

Example 24.3: The empirical formula of benzene is CH. It molecular mass is 78 amu. Determine its molecular formula.

Data:

Empirical formula of benzene	= CH
Empirical formula mass of benzene	= 13 amu
Molecular formula of benzene	= ?
Molecular formula mass of benzene	= 78 amu

Solution:

i) $\text{Value of } n = \frac{78\,\text{amu}}{13\,\text{amu}} = 6$

ii) Molecular formula of benzene = 6 (CH) = C_6H_6

24.2 Modern Methods of Analysis

These are the newer methods of chemical analysis. These methods use modern or highly specialized instruments to measure physical quantities of the analyte such as light absorption or emission, fluorescence, mass-to-charge ratio, or conductivity. Highly efficient chromatographic and electrophoretic techniques are used to replace the classical methods (distillation, extraction and precipitation) for the separation of components of complex mixtures. Examples of modern methods are spectroscopy, chromatography, microscopy, etc.

Modern methods are expensive because of the use of high specialized machines for chemical analyses. Modern methods have certain advantages over classical methods, which include the speed (they are fast), high sensitivity (they can detect very small amounts of a substance in a small amount of sample), versatility (simultaneous detection capabilities), multi-element analysis and automated operation of modern instruments.

In the early years of chemistry, the unsaturation in a compound was determined by the amount of hydrogen or halogen consumed. The presence of functional groups in organic compounds was determined by chemical tests. The halogens or sulphur in organic compounds were detected by chemical tests (sodium fusion tests). The aromaticity was determined by flame test. The molecular masses of compounds were determined by using the colligative properties.

Today's scientists have developed sophisticated instruments that enable chemists to:

i) Determine the unsaturation in a compound by UV spectroscopy.
ii) Determine the presence of functional groups in organic compounds by IR spectroscopy.
iii) Detect the halogens or sulphur in organic compounds by mass spectroscopy.
iv) Measure the aromaticity by NMR spectroscopy.
v) Determine molecular masses by mass spectroscopy.

Organic chemistry has been revolutionized by the invention of spectroscopic methods. Spectroscopic methods are used for analysis and structure determination.

The spectrometric methods use electromagnetic radiations as the basic tool. It is therefore necessary to understand the nature of electromagnetic radiations.

Electromagnetic Radiations

Electromagnetic radiation (EMR) is a form of energy and consists of both electric and magnetic fields. These fields are at right angles to each other and to the direction in which the wave is moving. Electromagnetic radiation carries energy through space, it is also called radiant energy.

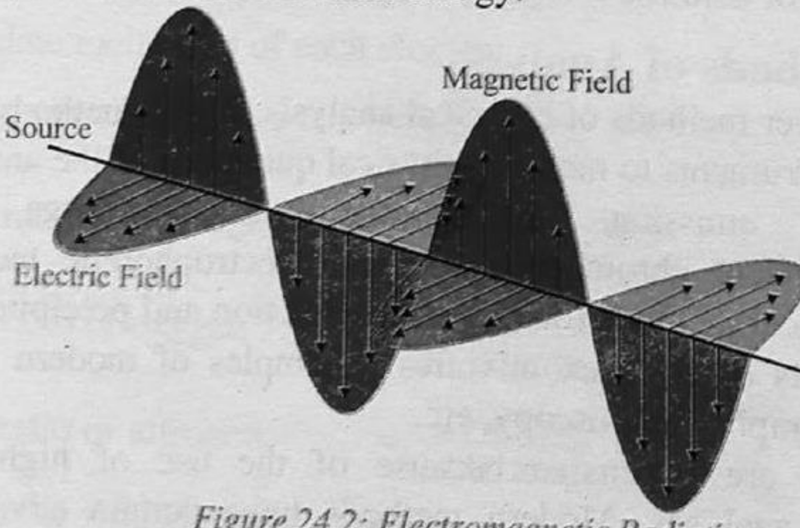


Figure 24.2: Electromagnetic Radiations

Electromagnetic radiations are all around us and take many forms. The heat (infrared radiations) from a burning fire, the light that is emitted from the bulb, sun or television, the X-rays used by doctors, the energy used to cook food in a microwave and the radio waves that carry music to our radios are all forms of electromagnetic radiation. Electromagnetic radiation includes gamma ray, X-ray, ultra-violet (UV), visible, infra-red (IR), microwave and radio wave. They seem very different from one another, but they all show wavelike properties. Their waves are similar to those waves that move through water. All types of electromagnetic radiations move through a vacuum with the velocity of light (2.998×10^8 m/s). All electromagnetic radiations travel in straight lines. They are not affected by electric or magnetic fields. All electromagnetic waves can be reflected, refracted and diffracted just like light waves. Visible light (the light we see with our eyes) is only a small portion of the EM spectrum, which contains a broad range of electromagnetic wavelengths.

Wavelength, Frequency and Energy

All electromagnetic radiations can be characterized by their wavelengths, energy and frequency. The energy of radiation increases with increasing frequency and decreases with increasing wavelength.

$$E = h\nu = hc/\lambda$$

Where, E is energy, h is Planck's constant, ν (nu) is frequency, c is velocity of light and λ (lambda) is wave length of radiations.

Keep in Mind

Wavelength

The distance between two adjacent crests or troughs of wave is called wavelength. It is shown by λ (lambda). Its units are m, nm or A°.

Frequency

The number of waves passing through a point in one second is called frequency. It is shown by 'ν'. Its units are hertz or cycles/sec.

$$\nu = c/\lambda$$

Frequency (ν) is inversely proportional to wavelength (λ).

Wavenumber

The number of waves present in one centimetre distance is called wave number. It is the inverse of wavelength. It is shown by $\bar{\nu}$ (nu bar). The unit of wavenumber is cm^{-1}.

Electromagnetic Spectrum

In addition to visible region, there are seven other regions of the spectrum. The ultraviolet, X-rays and γ-rays are towards the lower wavelength end and infrared, microwave and radio waves are towards higher wavelength end. Visible light falls in the range of the electromagnetic spectrum between infrared (IR) and ultraviolet (UV). It has wavelengths of about 400 nanometers (nm) to 800 nm. When the different types of electromagnetic radiations are arranged in order of their increasing wavelengths or decreasing frequencies, the complete arrangement is called electromagnetic spectrum. Although almost all parts of the electromagnetic spectrum are used for studying matter but in organic chemistry we are mainly concerned with three or four regions that are ultraviolet-visible, infrared, microwave and radio wave.

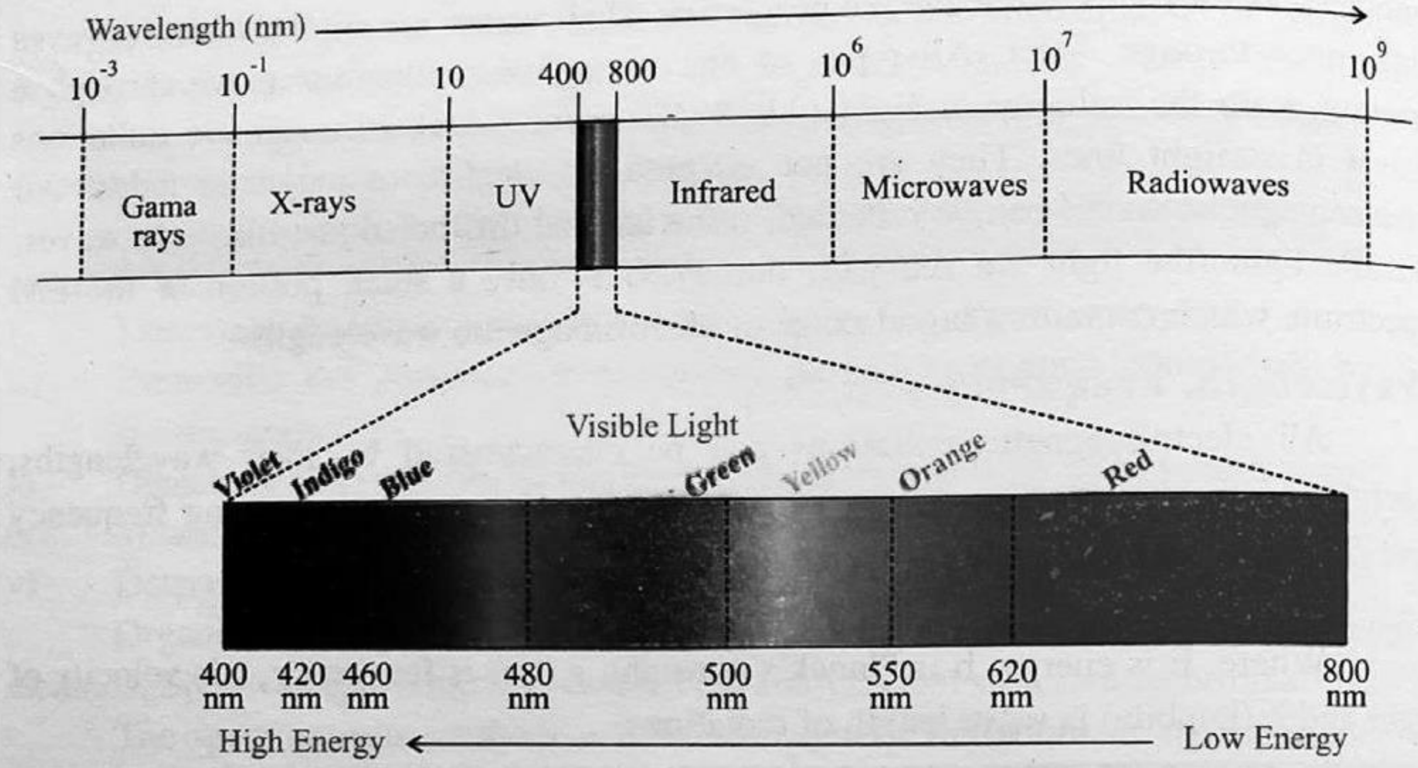


Fig. 24.3: Spectrum of Electromagnetic Radiations

The regions in increasing order of wavelength are:

Gamma Rays: the energy change involves the nuclear excitation.

X-rays: the energy changes involve the inner electrons of an atom or molecule.

UV-Visible: the energy changes involve valence electrons of molecules.

Infrared: the energy changes involve transitions between the vibrational levels of molecules.

Microwave: the energy changes involve transitions to higher energy rotational levels of molecules.

Radio wave: the energy changes involve the reversal of spin of a nucleus or electron.

Table 24.1: Electromagnetic Radiations and their Sources

Electromagnetic Radiations	Wavelength range (nm)	Sources	Uses
Gamma rays	10^{-3} — 10^{-1}	Sun, star, Unstable atomic nuclei	Used to kill cancer cells and may used for imaging.
X-rays	10^{-1} — 10^{1}	Heavy metal anode	Used to image internal bones and organs
UV-light	10 — 400	Sun light	Used to sterilize food, medicine and surgical equipments and cause the body to produce vitamin D.

Electromagnetic Radiations	Wavelength range (nm)	Sources	Uses
Visible light	400 — 800	Sun, light bulb	Enable us to see
IR light	800 — 10^6	Hot objects	Warm objects and used in night vision technology to "see" in the dark
Microwave	10^6 — 10^7	Oven, radar	Used for radar and in microwave oven to warm food
Radio wave	10^7 — 10^9	Radio, TV	AM and FM radio, TV, mobile phone

24.2.1 Spectroscopy

Spectroscopy is the branch of science which deals with the study of interaction of electromagnetic radiation with matter. It is one of the important experimental techniques used to determine the electronic structure of atoms and molecules. It is used to determine the structure and functional groups in organic compounds and is useful for studying the properties of molecules. It is used to monitor the progress of chemical processes. It can also be used to measure the purity of products. Spectroscopy is also used to determine the chemical composition and physical properties of astronomical objects (such as their temperature and velocity).

24.2.2 Spectroscopic Methods

The spectroscopic methods most often used by organic chemists are: ultraviolet (UV) and visible, infrared (IR), nuclear magnetic resonance (NMR) and mass spectroscopy (MS).

24.2.2.1 Ultraviolet-visible Spectroscopy

The spectroscopy which deals with the study of interaction of electromagnetic radiation with matter in the ultraviolet region (200-400nm) and visible region (400-800nm) is called ultraviolet-visible (UV/Vis) spectroscopy. The amount of energy available in this radiation is sufficient to cause the promotion of loosely held electrons (non-bonding electrons or the electrons involved in a π-bond) to higher energy levels. As UV/Vis spectroscopy involves electronic transitions, hence it is also known as electronic spectroscopy. Keep in mind that the energy levels of an atom are quantized, only light with specific amount of energy can cause transitions from one level to another.

Principle of UV Spectroscopy

Molecules containing π-electrons or non-bonding electrons can absorb energy in the form of UV-Visible light to stimulate these electrons to higher anti-bonding molecular orbitals. The more easily excited the electrons from lower energy level to higher energy level, the longer the wavelength of light it can absorb.

The greater is the number of the molecules that are capable of absorbing light at a certain wavelength, the greater is the extent of the absorption of light.

Types of Electrons Present in Compounds

In UV/Vis spectroscopy, three types of electrons are involved in transitions:

i) Sigma electrons (σ-electrons): these electrons are present in sigma orbitals. σ-Bonds are strong so that high energy is required for the promotion of these electrons. These types of electrons are found in alkanes.

ii) Pi-electrons (π-electrons): these electrons are present in π-orbitals. These electrons are found in those molecules which have double or triple bonds. Examples are alkenes, alkynes, aromatic compounds, etc.

iii) Non-bonding electrons (n-electrons): these electrons are not involved in bonding. Non-bonding electrons are found in those compounds, which have heteroatoms (N, O, S, etc.).

Types of Electronic Transitions

Four types of electronic transitions are possible:

(i) $\sigma \rightarrow \sigma^*$ (ii) $\pi \rightarrow \pi^*$ (iii) $n \rightarrow \sigma^*$ (iv) $n \rightarrow \pi^*$

(i) • $\sigma \rightarrow \sigma^*$ Transition

(ii) • $\pi \rightarrow \pi^*$ Transition

(iii) • $n \rightarrow \sigma^*$ Transition

(iv) • $n \rightarrow \pi^*$ Transition

The $\sigma \rightarrow \pi^*$ transition is not allowed by selection rules. The order of energy for different transitions is as: $n \rightarrow \pi^* < \pi \rightarrow \pi^* < n \rightarrow \sigma^* << \sigma \rightarrow \sigma^*$

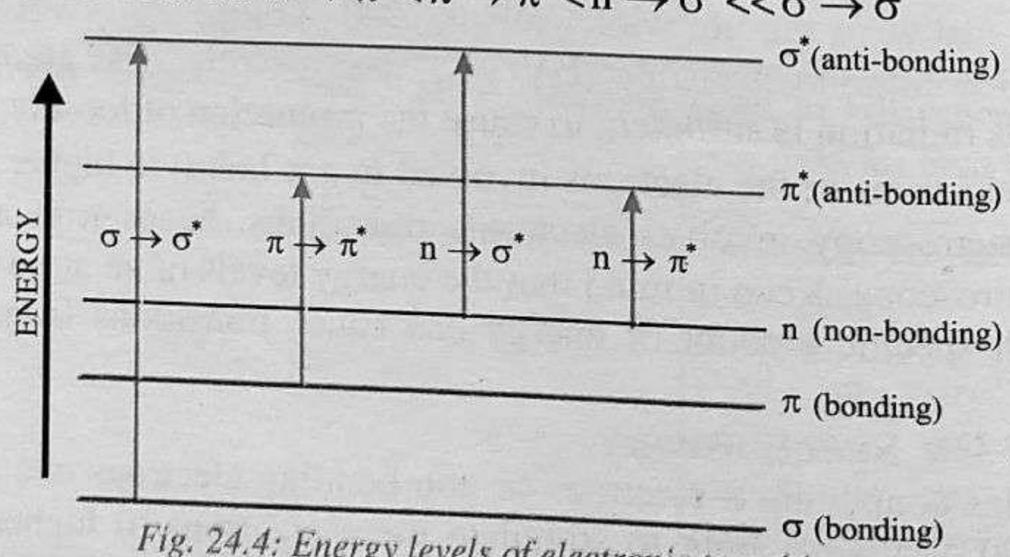


Fig. 24.4: Energy levels of electronic transitions

i) Sigma to Sigma Star ($\sigma \rightarrow \sigma^*$) Transitions

The promotion of electron from bonding sigma orbital to associated high energy antibonding sigma orbital ($\sigma \rightarrow \sigma^*$) require higher amounts of energy and usually involve absorption of ultraviolet radiation below 150nm. This is due to stronger sigma bonds. This is the only transition available in alkanes. For example, methane has carbon-hydrogen bonds only and shows absorbance maximum at 125nm. These transitions are not available in normal UV-Visible regions (200-800nm).

ii) Pi to Pi Star ($\pi \rightarrow \pi^*$) Transitions

The promotion of electron from bonding pi orbital to associated high energy antibonding pi orbital ($\pi \rightarrow \pi^*$) require lesser amounts of energy than $n \rightarrow \sigma^*$, $\sigma \rightarrow \sigma^*$ transitions. The transitions in unconjugated alkenes usually involve absorption of ultraviolet radiation around 170-190 nm and the non-conjugated carbonyl compounds involve the absorption of visible radiation around 200-300nm. This transition is available in conjugated alkenes, alkynes, carbonyls, nitriles, aromatic compounds, etc.

iii) n to Sigma Star ($n \rightarrow \sigma^*$) Transitions

The promotion of electron from non-bonding orbital to high energy antibonding sigma orbital ($n \rightarrow \sigma^*$) require lesser amounts of energy than $\sigma \rightarrow \sigma^*$ transitions and usually involve absorption of 150-250nm. This type of transition is shown by saturated compounds containing atoms with lone pairs of electrons like N, O, S and halogens. For example, the maximum absorption for hydrogen monoxide (H_2O) is 167nm and that of methyl alcohol (CH_3OH) is 183nm.

iv) n to Pi Star ($n \rightarrow \pi^*$) Transitions

The promotion of electron from non-bonding orbital to high energy antibonding pi orbital ($n \rightarrow \pi^*$) require the least amounts of energy than all other transitions and usually involve absorption at longer wavelengths around 300nm. This kind of transition is shown by unsaturated molecules containing heteroatoms such as N, O and S. Saturated aliphatic ketones show absorption around 280nm. For example, acetone shows absorption maximum at 279 nm.

Instrumentation and Working of UV/Visible Spectroscopy

The modern UV spectrometers (double beam spectrophotometer) consist of the following parts:

i) Light source
ii) Monochromator
iii) Beam Chopper
iv) Sample and reference cells
v) Detectors
vi) Amplifier
vii) Recording device

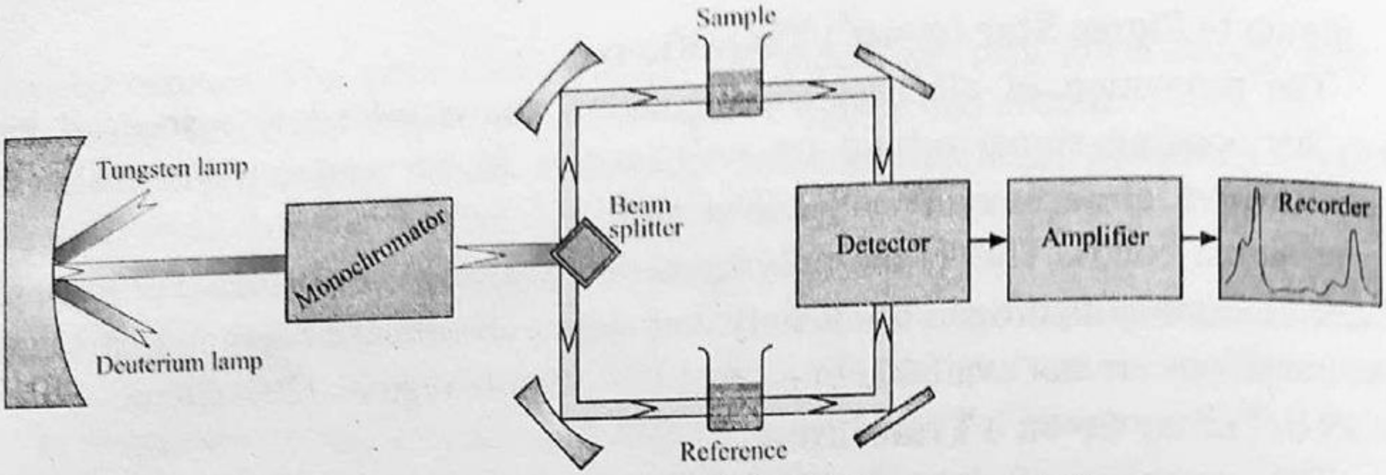


Fig. 24.5: Instrumentation of UV/Visible spectroscopy

Light Source

. Tungsten filament lamps and Hydrogen-Deuterium lamps are most widely used.

Monochromator

Electromagnetic radiation from the source of light enters the monochromator through entrance slit. The monochromator is used to disperse the radiation into different wavelengths. Monochromators (also known as wavelength selector) generally composed of prisms and slits.

Beam Chopper (Beam Splitter)

Chopper is device consisting of circular disc. It splits the monochromatic beam of light into two beams of equal intensities.

Sample and reference cells

These cells are made of either silica or quartz. Sample under measurement and the reference solution are placed in the cells. One of the two divided beams is passed through the sample solution and second beam is passed through the reference solution.

Detectors

Detectors have photocells. One of the detector receives the beam from sample cell and second detector receives the beam from the reference. Both the detectors generate voltage proportional to the radiation energy that strikes them.

Amplifier

The voltage generated in the photocells is transferred to the amplifier. Generally voltage generated in the cells is of very low intensity, the main function of amplifier is to amplify the signals many times to generate clear and recordable signals.

Recording device

Recording device is generally computer system. It automatically stores and records all the data and generate the spectrum of sample under investigation as a plot of wavelengths of absorbed radiations against absorbance.

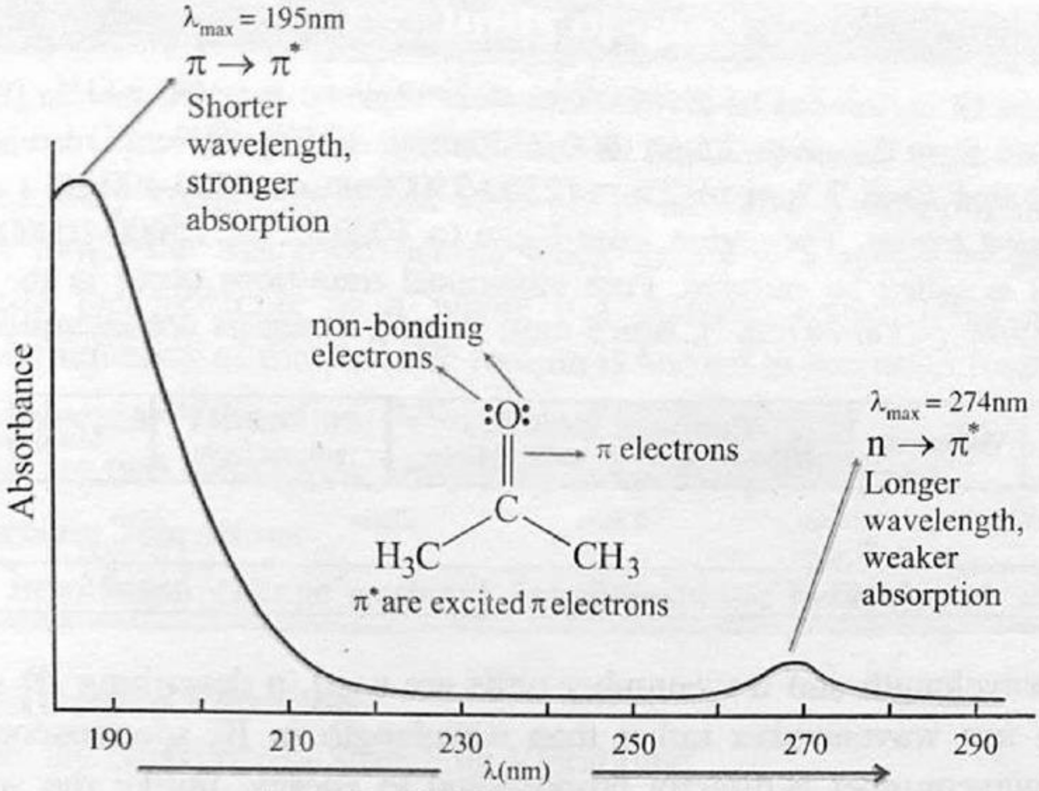


Fig. 24.6: UV/Vis Spectrum of Acetone

Applications of UV-Visible Spectroscopy

UV-Visible spectroscopy is one of the most useful tools and is usually used in analytical chemistry for qualitative and quantitative analyses. Some of the applications of UV-Visible spectroscopy are:

i) It is used for the determination of equilibrium and dissociation constants.

ii) It is useful for the determination of rate of chemical reaction.

iii) It is also useful to detect the structure of organic compounds, the presence or absence of unsaturation, the presence of heteroatoms and the impurities in the samples of organic compounds.

24.2.2.2 Infrared Spectroscopy

The spectroscopy which deals with the study of interaction of electromagnetic radiation with matter in the infrared region from 0.8μm to 1000μm (or 800-1000000nm) is called infrared (IR) spectroscopy. IR spectroscopy is concerned with the study of absorption of infrared radiation, which causes vibrational transition in the molecule. Hence, IR spectroscopy is also known as vibrational spectroscopy. Not all the molecules can absorb IR radiation. Molecules absorb radiation when a bond in the molecule vibrates at the same frequency as the incident radiant energy. After absorbing energy, the molecules have more energy and vibrate at increased amplitude.

Keep in Mind

The entire IR region can be divided into three regions: near IR, middle IR and far IR. The region from 0.8μm to 2.5μm (800-2500nm or 12500-4000cm^{-1}) is called near infrared and that from 2.5μm to 25μm (2500-25000nm or 4000-400cm^{-1}) is called middle infrared region. The region from 25μm to 1000μm (or 25000-1000000nm or 400-10cm^{-1}) is called far infrared. Pure vibrational transitions occur in the infrared region 2.5-15μm (4000-667cm^{-1}), hence most organic chemists are interested in this region.

	Visible Light	Near Infrared Light	Middle Infrared Light	Far Infrared Light	Microwaves	
Wavelength	400nm	800nm (0.8μm)	2.5μm	25μm	1mm (10^6nm)	10^7nm
Wavenumber		12500cm^{-1}	4000cm^{-1}	400cm^{-1}	10cm^{-1}	

Both wavelength and wavenumber units are used in describing IR spectra but most chemist use wavenumber rather than wavelength in IR spectroscopy. This is because the wavenumber is directly proportional to energy, unlike the wavelength, which is inversely proportional to energy. Thus, the wavenumber is more comparable to frequency as compared to wavelength.

Keep in Mind

When we sit in the sun or close to a heater or fire, we are irradiated by infrared radiations (also called heat waves) of the spectrum. The energy provided by this type is enough to vibrate our skin molecules, this vibration increases the kinetic energy of our skin molecule, so we feel warmth.

Principle of IR Spectroscopy

Molecules are made up of atoms linked by chemical bonds. The movement of atoms and the chemical bonds is like spring and balls (vibration).This characteristic vibration is called natural frequency vibration.If the bond is stretched or compressed and then released, the atoms vibrate.

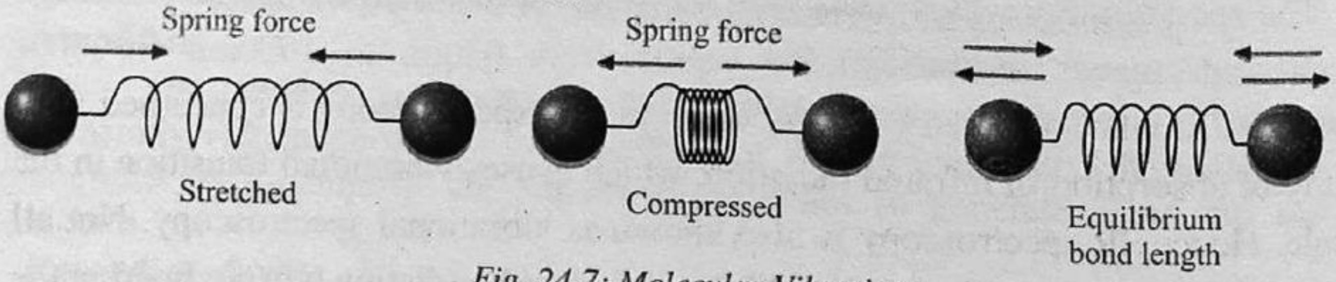


Fig. 24.7: Molecular Vibrations

When energy in the form of IR radiation is applied then it causes the vibration between the atoms of the molecules. As a result the absorption of IR radiation takes place and a peak is observed.

Molecular Vibrations (Vibrational Modes) of IR Spectroscopy

Atoms in a molecule do not remain at fixed positions with respect to each other, but actually vibrate back and forth about an average value of the interatomic distance. A molecular vibration occurs when atoms in a molecule are in periodic motion while the molecule as a whole has constant translational and rotational motion. The frequency of the periodic motion is known as vibration frequency.

Types of Molecular Vibrations (Vibrational Modes)

There are two main types: stretching and bending vibrations.

i) Stretching Vibrations

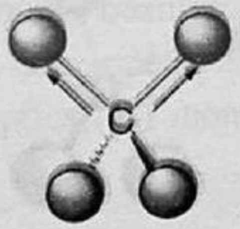
Stretching involves a change in bond lengthsresulting in a change in interatomic distance.

Types of Stretching Vibration

There are two types of Stretching Vibrations:

Symmetric stretching:

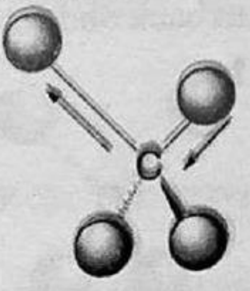
In symmetric stretching vibration, two or more bonds vibrate in and out together.



Symmetric Stretching

Asymmetric stretching:

In asymmetric stretching, some bonds are getting shorter and the others are getting longer at the same time with respect to the central atom.



Asymmetric Stretching

ii) Bending vibrations

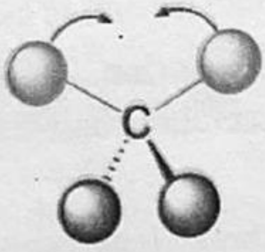
Bending vibrations involve the change in bond angles or change in the position of a group of atoms with respect to the rest of the molecule. The bond lengths, in bending vibrations, remain constant.

Types of Bending Vibrations

There are four types of bending vibrations:

Scissoring

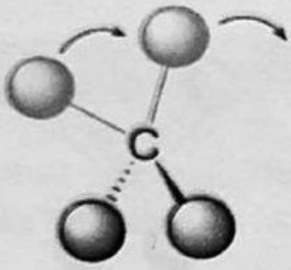
Scissoring is the movement of two atoms toward and away from each other like the movement of scissors.



Scissoring

Rocking

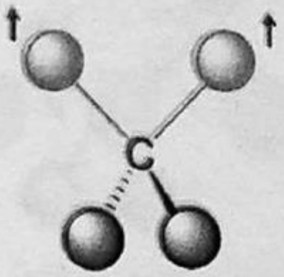
Rocking is the movement of atoms in the same direction within a plane. It is like the motion of pendulum on a clock, here an atom is a pendulum and there are two instead of one.



Rocking

Wagging

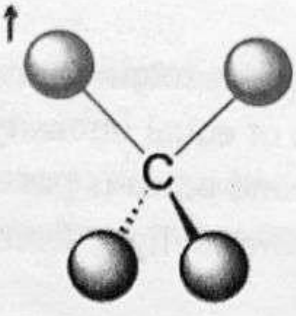
Wagging is the simultaneous motion of two atoms that move up and down the plane with respect to the central atom. It is like the motion in which you make a "V" sign with your fingers and bend them back and forth from your wrist.



Wagging Vibrations

Twisting

Twisting involves when one atom moves up the plane and the other atom moves down the plane with respect to the central atom.



Twisting Vibrations

Factors Affecting Vibrational Frequency

The frequency absorbed depends on:

(i) the masses of atoms in the bond (ii) the strength of the bond (iii) the geometry of the molecule (iv) and several other factors.

Keep in mind that heavier the atoms, lower is the vibration frequency of the bond between them. Stronger the bond, higher is the vibration frequency.

Instrumentation of IR Spectroscopy

Instrument that is used to determine the absorption spectrum of compound is called infrared spectrometer or, more accurately, infrared spectrophotometer. The most common spectrophotometer is double beam spectrophotometer. It is similar to that of UV spectrophotometer. Its main components are:

(i) radiation Source (ii) Fore optics (iii) monochromator (iv) sample cell (v) detector (vi) recorder

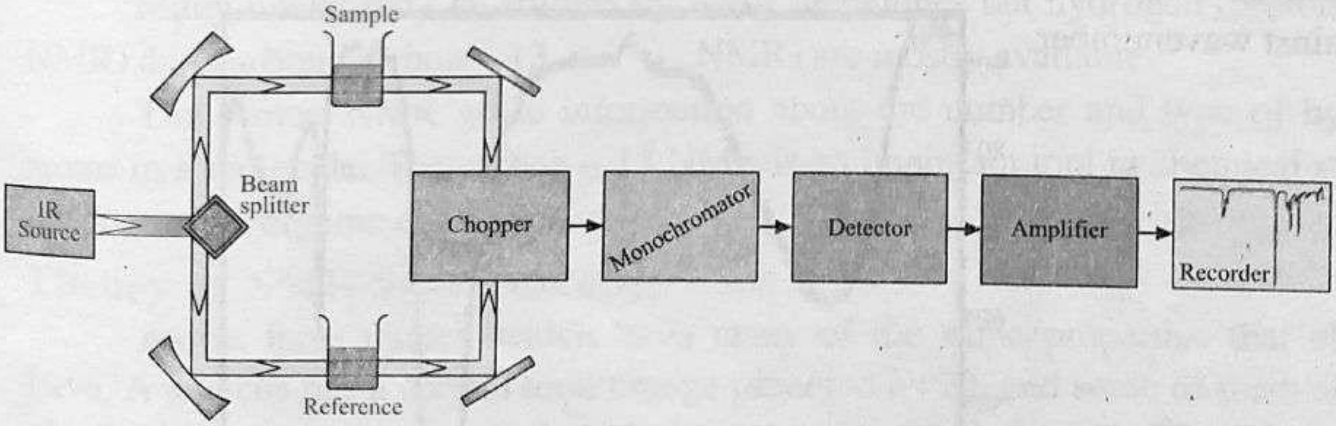


Fig. 24.8: IR Spectrophotometer

i) Radiation Source

The commonly used sources are Nernst glower and globar.

Nernst glower is ahollow rod of rare earth oxides (zirconium, thorium and cerium).

Globar is a silicon carbide rod.

Nernst glower is used for shorter wavelengths and globar filament is used for longer wavelengths.

ii) Optics

Optics consists of mirrors and a rotating mirror. Mirrors divide the light from the infrared source into two beams of equal intensity. One of the two divided beams is passed through the sample and second beam is passed through the reference (solvent). The rotating mirrors alternately allows light from each of the beams to enter the monochromator.

iii) **Chopper**

Chopper recombines the light before entering into monochromator. It passes the two beams alternately to a prism (or diffraction grating).

iv) **Monochromator**

Prisms and grating can be used as monochromator. Monochromator produces single wavelength radiations.

v) **Detector**

Detectors convert thermal energy into electrical energy.

vi) **Amplifier**

The signals obtained in the detector are amplified many times to generate clear and recordable signals.

vii) **Recorders**

Recorder automatically records all the data on a computer and plots the IR spectrum of the sample on a graph. It usually plots the transmittance or absorbance against wavenumber.

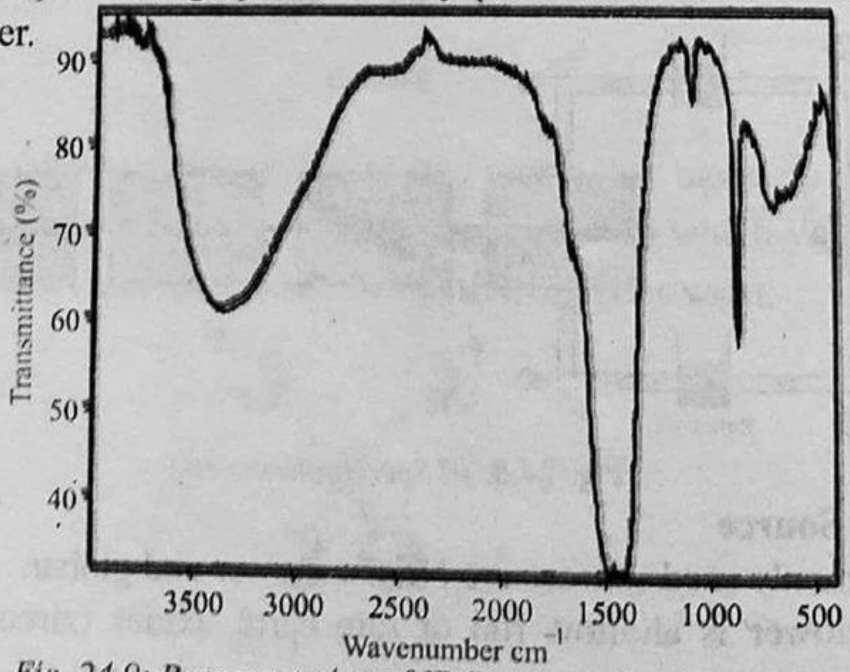


Fig. 24.9: Representation of IR Spectrum of a Sample

Keep in Mind

The frequency is usually plotted as transmitted light, nor light absorbed.

Applications of IR Spectroscopy

It is widely used in both organic and inorganic chemistry. Some of the applications of IR spectroscopy are:

i) It is mainly used for structural determination of organic compounds.

ii) It is used to detect the presence or absence of a particular functional group in the compound.

iii) It is being used to help identify the structure of complex molecules in space.

iv) It is very effective for the detection and measurement of even minute quantities of the chemical compounds; hence it is widely applied in environment analysis, forensic analysis, industries and research.

v) It has also been successfully utilized in the field of semiconductor microelectronics: for example, infrared spectroscopy can be applied to semiconductors such as silicon, gallium arsenide, gallium nitride, zinc selenide, amorphous silicon, silicon nitride, etc.

24.2.2.3 Nuclear Magnetic Resonance (NMR) Spectroscopy

The spectroscopy which deals with the study of interaction of electromagnetic radiation with matter in the radiofrequency region is called nuclear magnetic resonance (NMR) spectroscopy.

The range of NMR region is from 7.5×10^{10} nm to 4.0×10^{8} nm (75m to 0.4m or 4 MHz to 750 MHz).

Many nuclei may be studied by NMR techniques but hydrogen (proton i.e. ^{1}H NMR) and carbon (carbon – 13 i.e. ^{13}C NMR) are mostly available.

The proton NMR gives information about the number and type of hydrogen atoms in a molecule. The carbon – 13 NMR is an important tool in chemical structure elucidation in organic chemistry.

Theory of NMR Spectroscopy

Atoms have nuclei, which have many of the same properties that electrons have. A nucleus has a certain total charge (denoted by Z), and some of them also spin about their axes in a manner that electrons spin about their axes. The spin quantum number (spin) of a nucleus is denoted by ℓ. The spin quantum number of a nucleus is determined by the number and pairing of the individual particles. The rules for determining the net spin of a nucleus are as:

i) The nucleus has **NO** spin when the number of protons and the number of neutrons are both even.

ii) The nucleus has a half-integer spin (i.e. 1/2, 3/2, 5/2) when the sum of the number of protons and neutrons is odd.

iii) The nucleus has an integer spin (i.e. 1, 2, 3) when the sum of the number of protons and neutrons is even. The number of protons and the number of neutrons are both odd in this case.

Table 24.2: The Spin Quantum Number of Some Atoms

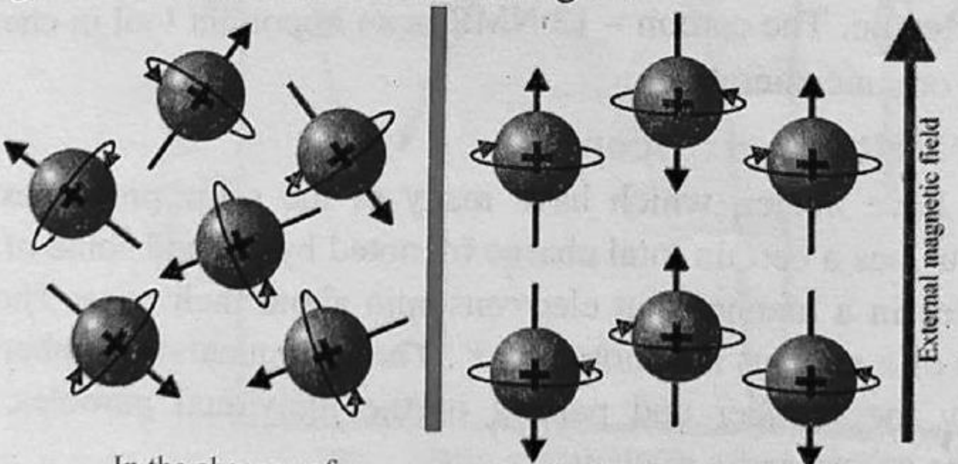
No. of Protons (Atomic Number)	No. of Neutrons	Mass Number	Spin Quantum Number	Examples
Even	Even	Even	Zero	$^{4}_{2}$He(0), $^{12}_{6}$C(0), $^{16}_{8}$O(0)
Even or odd	Even or odd	odd	Half-integer	$^{1}_{1}$H(1/2), $^{7}_{3}$Li(3/2), $^{13}_{6}$C(1/2), $^{17}_{8}$O(5/2)
Odd	Odd	Even	Integer	$^{2}_{1}$H(1), $^{6}_{3}$Li(1), $^{10}_{5}$B(3) $^{14}_{7}$N(1)

The table shows that the nuclei with an odd mass number or odd atomic number have nuclear spins. The nuclei that have nuclear spins such as ^{1}H or ^{13}C are said to be NMR active nuclei i.e. they have magnetic fields. When the nuclei have an even number of protons and neutrons such as ^{12}C and ^{16}O, their magnetic fields cancel out and they have no overall magnetic fields. These nuclei are NMR inactive.

Principles of NMR Spectroscopy

All nuclei with non-zero spins have magnetic moments (μ). Thus the nucleus can be considered to be a tiny bar magnet. The magnetic moment is generated by charge and spin of nuclei.

Without an external applied magnetic field, the nuclear spins are random in directions. But when an external magnetic field is applied, the nuclei align themselves either with or against the field of the external magnet.



In the absence of magnetic field

In the presence of magnetic field

Fig. 24.10: Nuclear Spin

It is possible to excite these nuclei into the higher level with electromagnetic radiation. The frequency of radiation needed is determined by the difference in energy between the energy levels.This is very important to know that the lower energy level will contain slightly more nuclei than the higher level.

In the presence of an external magnetic field, two spin states exist, +1/2 and −1/2.

The magnetic moment of the lower energy +1/2 state is aligned with the external field, but that of the higher energy −1/2 spin state is opposed to the external field.

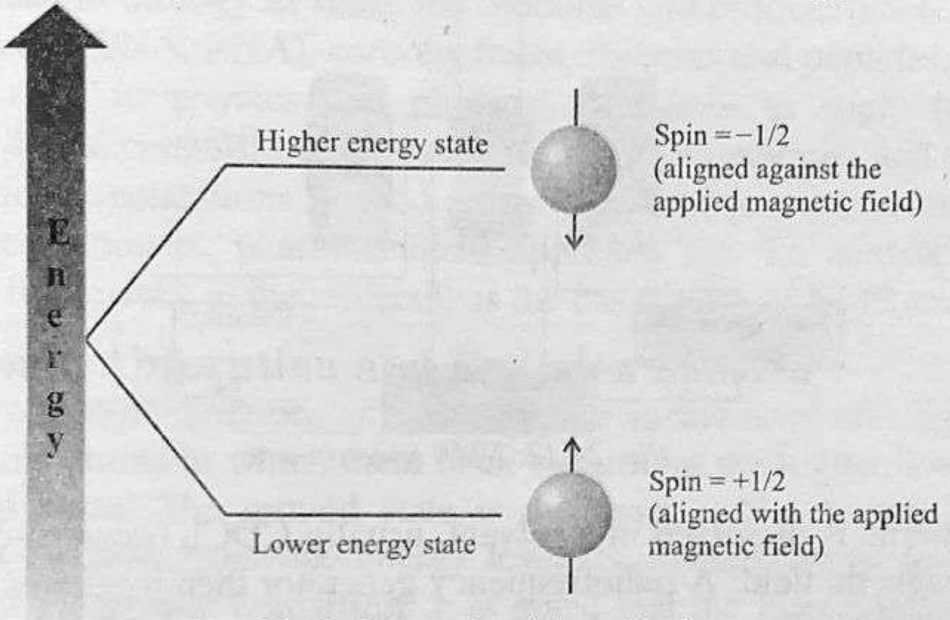
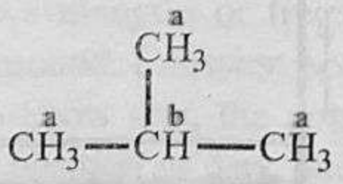


Fig. 24.11: Energy Level for a Nucleus

The stronger the magnetic field, the larger the energy difference between two nuclear spin states and higher the energy (frequency, ν) needed for the resonance.

The amount of energy absorbed is recorded as NMR Spectrum. In practice either the magnetic field can be held constant and the radio frequency varied or more commonly the radio frequency can be held constant and the magnetic field varied. The main purpose of NMR is not to detect the presence of proton in a molecule. It can be distinguish between protons in different chemical environments with in the molecule. The number of signals recorded on the NMR chart paper indicates the number of different types of protons in molecules. The position of the peak can give information about the molecule structure in the vicinity of the proton. Protons are of two types' equivalent and non-equivalent protons. Equivalent protons are those in the molecule that are chemically indistinguishable while non-equivalent protons are chemically distinguishable.

$\overset{a}{CH_3}—\overset{b}{CH_2}—Cl$



$\overset{a}{CH_3}—\overset{\overset{a}{CH_3}\,|\,b}{CH}—\overset{a}{CH_3}$

a = Equivalent Protons
b = Non-equivalent Protons

Instrumentation and Working of NMR Spectrometer

Instruments that are used to get NMR spectra are usually named NMR spectrometer. It contains a complex collection of electronic equipment. NMR spectrometers use very powerful magnets to create a small but measurable energy difference between two possible spin states.

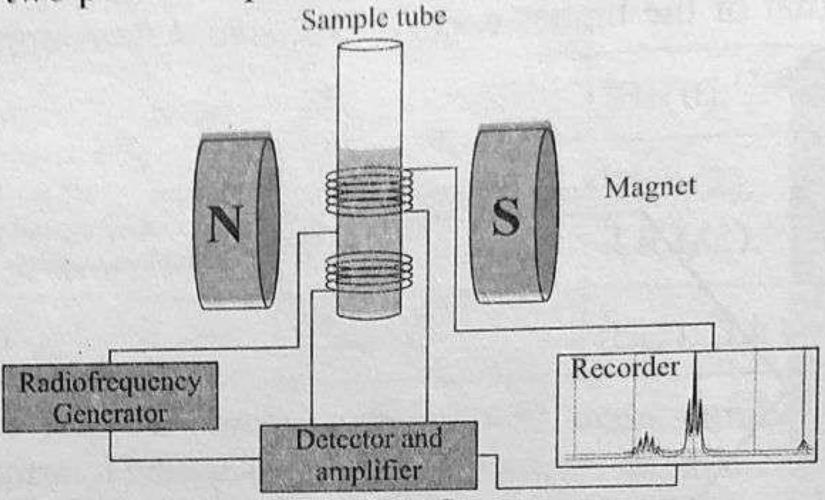


Fig. 24.12: NMR Spectrometer

Working

The sample is dissolved in a solvent, usually $CDCl_3$ (deutero-chloroform), and placed in a magnetic field. A radiofrequency generator then irradiates the sample with a short pulse of radiation, causing resonance. When the nuclei fall back to their lower energy state, the detector measures the energy released and a spectrum is recorded.

NMR Spectrum

The NMR spectrum is a plot of intensity of NMR signals versus magnetic field (frequency) in reference to TMS (tetramethylsilane). NMR signal is plotted on the y-axis and frequency on the x-axis. The value of frequency increases from right to left rather than left to right. In NMR spectrum we do not use unit Hertz (cycles per second) but rather we use parts per million (ppm).

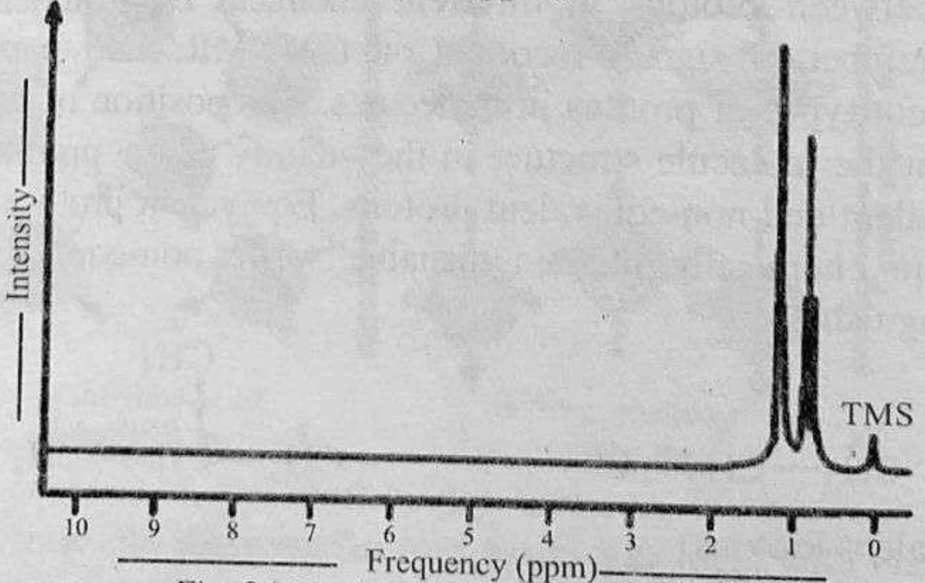


Fig. 24.13: A sample of NMR Spectrum

Applications of NMR Spectroscopy

Nuclear magnetic resonance (NMR) spectroscopy is one of the most powerful and widely used techniques in chemical research for investigating structure and dynamics of molecules. Some of the applications of NMR spectroscopy are:

i) Advanced methods can even be utilized for structure determinations of biopolymers, for example proteins or nucleic acids. NMR is also used in medicine for magnetic resonance imaging (MRI).
ii) NMR is used in biology to study the biofluids and biomacromolecules such as Nucleic acids (DNA, RNA), carbohydrates, Proteins and peptides.
iii) NMR is used in physics and physical chemistry to study high pressure diffusion, liquid crystals, liquid crystal solutions, membranes and rigid solids.
iv) It has various applications in food industries, food science, chemical analysis of different products, pharmaceutical approach etc. To analyse the carbon-hydrogen framework in the molecule is the basic work of NMR technique.

24.2.2.4 Atomic Absorption and Emission Spectra

Atoms in which no electrons are in the higher vacant level are said to be in the **ground state** while atoms in which there is an electron in the higher level are said to be in an **excited state**. The ground state is the most stable electronic state. The movement of an electron from one energy level to another energy level within an atom is known as **electronic transition**. It is noted that the light coming is must be exactly equal to the energy difference between the two electronic energy levels, otherwise the atom will not absorb it.

Atomic Absorption Spectrum

The absorption spectrum of an atom is the spectrum of wavelengths or frequencies of photons that are absorbed by the atoms due to jumping of electron from lower energy level to higher energy level. The wavelengths or frequencies that are absorbed will appear as dark lines on a bright background. Absorption occurs upon supplying radiation. The absorption spectrum of sodium gives two dark lines of wavelength 589.0 nm and 589.6 nm and that of hydrogen gives four different dark lines of wavelength 410 nm, 434 nm, 486 nm, and 656 nm.

Atomic Emission Spectrum

The emission spectrum of an atom is the spectrum of wavelengths or frequencies of photons that are emitted by the atom due to falling of electron from higher energy level to lower energy level. The wavelengths or frequencies that are emitted will appear as bright lines on a dark background. Emission occurs even in the absence of a radiation source. It is interesting to know that, the positions of bright lines in emission spectrum is exactly same as the position of dark lines in absorption spectrum, that is the energy of radiations emitted by excited hydrogen are exactly the same as energies absorbed by cold hydrogen. The emission spectrum of sodium gives two bright lines (golden yellow lines) of wavelength 589.0 nm and 589.6 nm and that of hydrogen gives four different bright lines of wavelength 410 nm (violet), 434 nm (blue), 486 nm (bluish-green), and 656 nm (red).

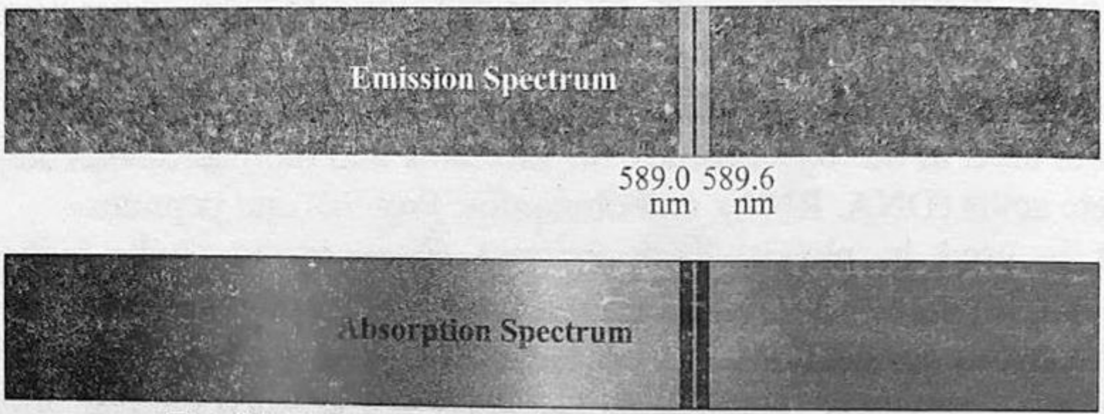


Fig. 24.14: Emission and Absorption Spectra of Sodium

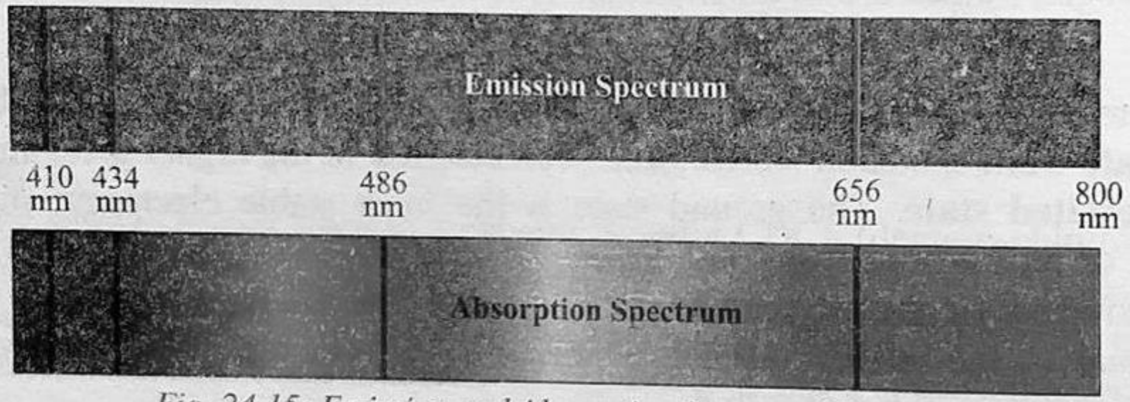


Fig. 24.15: Emission and Absorption Spectra of Hydrogen

Interesting Information

Strontium gives red, barium gives green, potassium gives violet and copper gives blue coloured lines on heating. These are the colours of the flame test of these elements. In the discharge tube, the helium gas glows with pink colour while neon glows with an orange red colour.

Elements can be identified by their spectral lines because the spectral lines are different for every element.

Table 24.3: Difference between Emission and Absorption Spectra

S.No.	Emission Spectrum	Absorption Spectrum
1	It is formed at high temperature.	It is formed at low temperature.
2	It is formed when the substance is in the gaseous state (vapours).	It is formed when the substance is even in the liquid or solution state.
3	In this spectrum, bright (coloured) lines are present on dark background.	In this spectrum, dark lines are present on bright background.

S.No.	Emission Spectrum	Absorption Spectrum
4	It has bright (definite colour) lines.	It has dark black lines.
5	It is formed when an electron jumps from higher energy level to lower energy level.	It is formed when an electron jumps from lower energy level to higher energy level.
6	Energy is released.	Energy is absorbed.

24.2.2.5 Mass Spectrometry (MS)

Mass spectroscopy is a technique that is used to measure molecular masses, to analyze the composition of sample and to elucidate the structures of molecules and other compounds by means of the separation of gaseous ions according to their mass-to-charge ratio (m/z). Mass spectroscopy is also known as mass spectrometry.

Mass spectroscopy is not a true spectroscopic technique because absorption of electromagnetic radiation is not involved at all. Mass spectroscopy has very high sensitivity, low detection limits and the possibility of ionizing very small sample volumes.

Principle of Mass Spectrometry

Mass spectrometry does not directly determine mass; it determined the mass-to-charge ratio. Mass spectrometry carry out three main functions that is: (i) the ionization of molecules, (ii) the separation of these molecules according to their mass-to-charge (m/z) ratio (iii) the determination of the respective abundance of each ion produced.

Instrumentation and Working of Mass Spectrometry

The simplest form of mass spectrometer has following components:

i) Sample inlet system (vapourizer)
ii) Ion source (ionization chamber)
iii) Accelerating plates and slits
iv) Analyzer (ion separation system)
v) Detector (ion collector)
vi) Amplifier
vii) Recorder

These basic parts are well shown in the following schematic diagram.

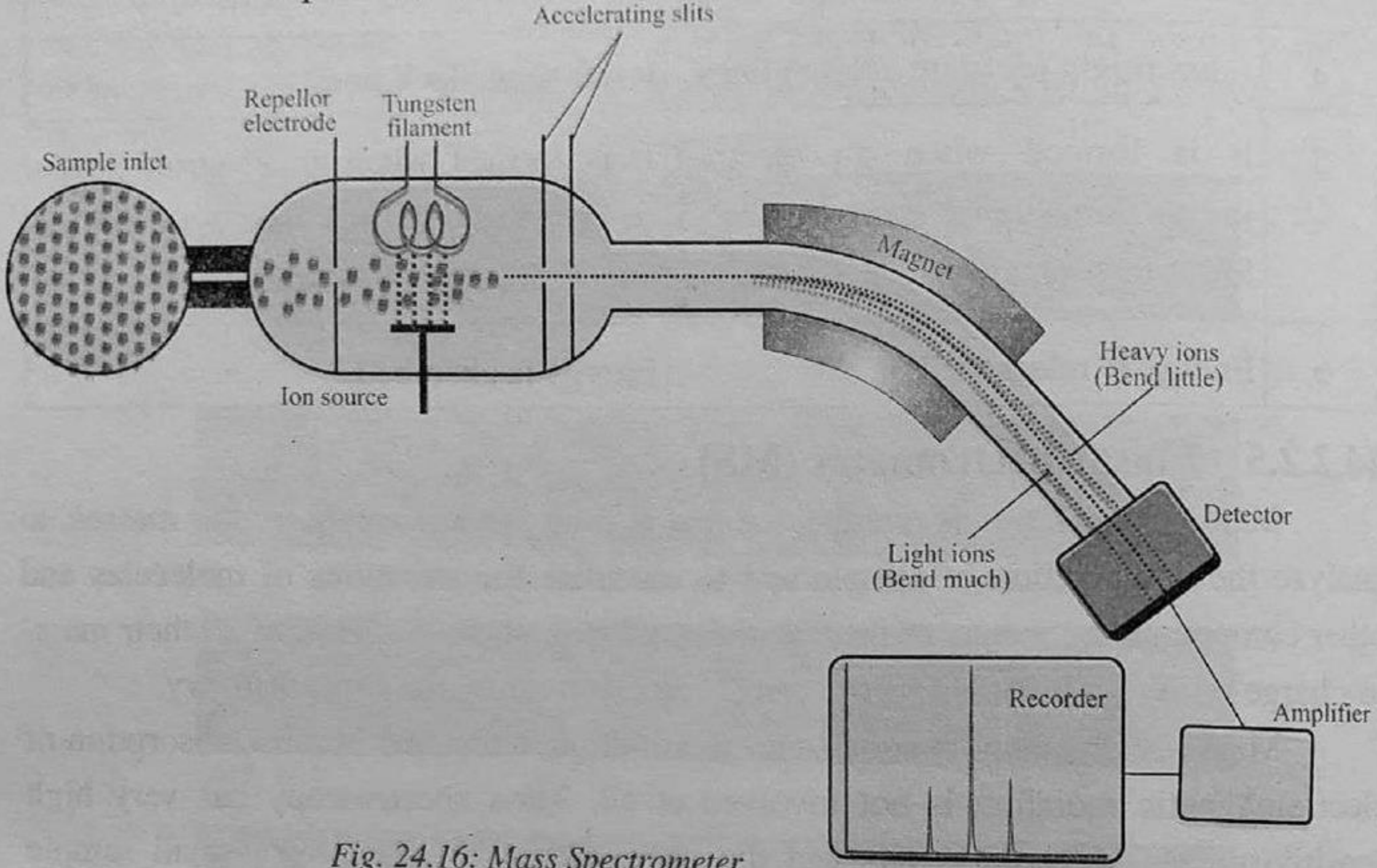


Fig. 24.16: Mass Spectrometer

The sample (material to be analysed) is introduced into an inlet chamber and converted into vapours state. These vapours are allowed to enter into ionization chamber.

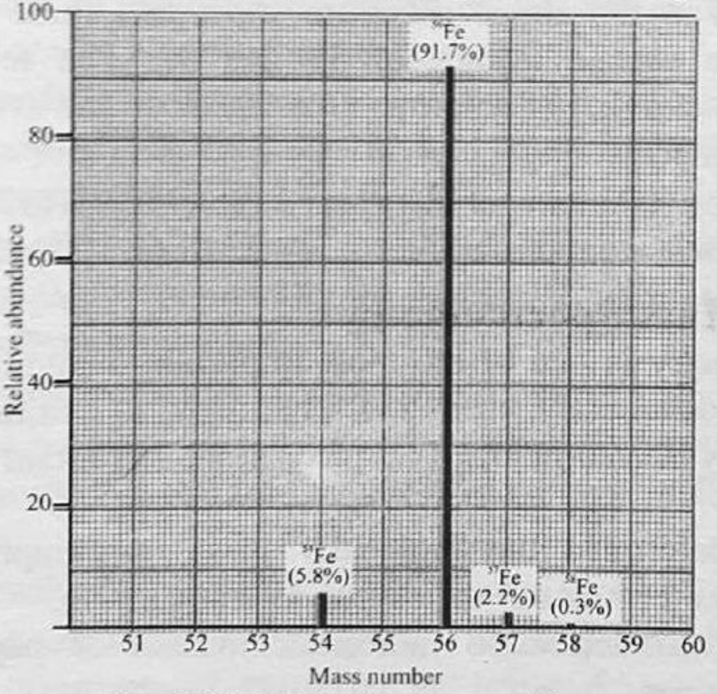
The ion source is an essential component of all the mass spectrometers where the sample molecules pass through a beam of very high energy electrons emitted from electrically heated filament (usually tungsten filament). The electron-molecule collision gives considerable energy to the sample molecules that in turn emit electrons and produce positively charged ions. These positively charged ions have different masses depending upon the nature of isotopes of the element present in them.

One essential component of all mass spectrometers is the vacuum system. It produces the required vacuum conditions for ion generation and for separation and detection of ions. The pressure of the system is kept at very low so the ions move freely without any resistance from air molecules.

A repeller plate, which carries a positive electrical potential, directs these newly created positive ions toward a series of accelerating plates. A large potential difference of 1 to 10 kilovolts (kV) is applied across the accelerating plates to produce a beam of fast moving positive ions. The positive ions pass through one or more focusing slits to produce a uniform beam.

Analyser (the ion separation system) separates the fast moving ions with respect to their mass-to-charge (m/z) ratios. If all ions have single positive charge and have the same velocity, then the amount of deflection is inversely proportional to the mass of each ion. The lighter ions are deflected more than the heavier ions.

The ions ultimately reach an ion collector, where they are detected. An electronic signal is generated by the collector and is amplified before it is recorded. Frequently the data go directly into an on-line computer which generates the mass spectrum in digital or bar graph form. The mass spectrum is the 2D representation of ion intensity (relative abundance) against the mass-to-charge (m/z) ratio. In mass spectrum, the highest peak, called the base peak, is the standard against which the other peaks are measured. The base peak is arbitrary assigned a relative intensity value of 100. The spectrum helps us in determining the relative atomic masses as well as relative abundance (ion intensity) of isotopes of elements. In mass spectrum, the position of peaks shows relative atomic mass and the length of peaks shows the relative abundance of the ion. Consider the example of mass spectrum of iron.



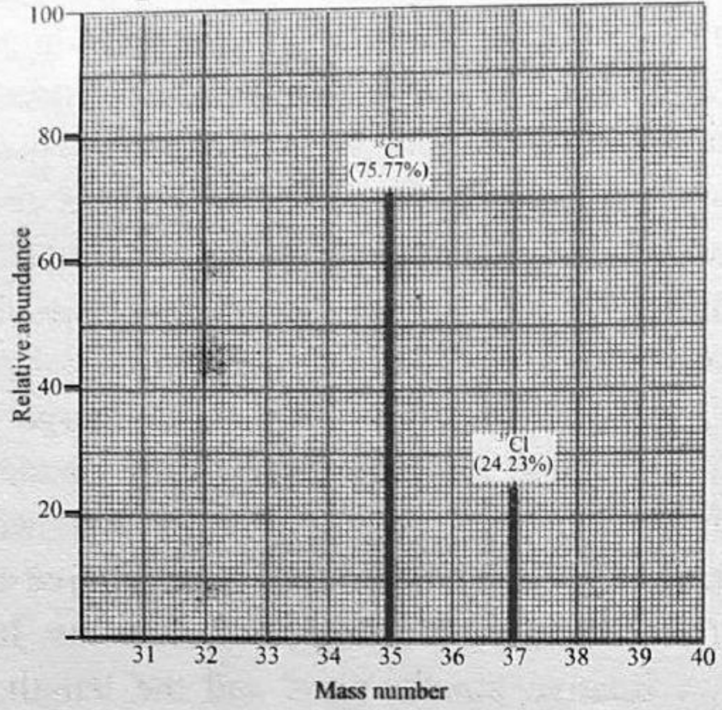
Graph 24.1: Mass Spectrum of Iron

Here the horizontal axis shows the mass of each detected ion. The relative abundance of each ion can be found from the height of each peak. It is seen that there exist four isotopes of Iron, i.e. ^{54}Fe, ^{56}Fe, ^{57}Fe and ^{58}Fe, each with a relative abundance 5.8%, 91.7%, 2.2% and 0.3% respectively. Thus ^{56}Fe is much more abundant in nature than the other isotopes. From this data the relative atomic mass of the 'Fe' is calculated as follows,

Average mass of 100 atoms of Fe $= (54\times5.8) + (56\times91.7) + (57\times2.2) + (58\times0.3) = 5591$

Average mass of 1 atom of Fe $= 5591.2/100 = 55.91$ amu

Consider another example of mass spectrum of chlorine.



Graph 24.2: Mass spectrum of chlorine

The two peaks in the above spectrum show that there are two isotopes of chlorine with relative atomic masses of 35 and 37. The relative abundance of chlorine-35 is 75.77% and that of chlorine-37 is 24.23%. The average atomic mass of chlorine can be calculated as:

Average mass of 100 atoms of chlorine $= (35\times75.77)+(37\times24.23)=3548$
Average mass of 1 atom of chlorine $= 3548.46/100 = 35.48$ amu

Applications of Mass Spectrometry

Mass spectrometry is one of the most important analytical techniques that is used in chemistry, biochemistry, medicine, pharmacy, agriculture, material science, food science and many related fields of science. Some important applications of mass spectroscopy are as:

i) It is used to determine the molecular mass of compounds by separating molecular ions on the basis of their mass-to-charge ratio.
ii) It is also used to determine molecular masses of peptides, proteins and oligonucleotides.
iii) It is used for characterization and sequencing of different proteins.
iv) It is used in materials science to identify the precise chemical composition of samples, which can help to explain why buildings or bridges collapse unexpectedly or why engineering components have suddenly failed.
v) It is used in forensic science to identify unusual substances found at a crime scene and match them precisely with similar substances found elsewhere.
vi) It is used to identify chemical, biological, radiological, nuclear, and explosive threats.

vii) It can help us to understand our environment. We can use them to measure how quickly air and water pollution travels.

viii) Mass spectrometers are increasingly being used for things like drug testing and discovery, food contamination detection, virus and bacteria identification, pesticide residue analysis, isotope ratio determination, protein identification, and carbon dating.

Summary of Facts and Concepts

- The analytical chemistry deals with the analysis of matter. The analysis means to find out the chemical composition of the matter that is, what type of atoms constitutes the matter and how many atoms are forming that matter.
- The traditional methods of analysis are called classical methods of chemical analysis which are based on using the chemical reactions for analytical procedures. The traditional methods are time consuming and needs a lot of expertise.
- The modern methods of analysis are based on the use of different radiations. These analytical procedures are termed as the spectroscopy.
- The modern methods called spectroscopy are fast, require less amount of substance to be analyzed, and they use modern equipment like computers which make the results more accurate.
- The matter interact with the light or radiations and as result of this energy is absorbed and subsequently emitted by the electrons in the atoms and molecules of any substance under observation.
- The table of all known radiations is called electromagnetic spectrum.
- The Instrument that is used in spectroscopy is called the spectrometer.
- Mostly used radiations of spectrum include infrared rays, visible light, ultraviolet rays and the X-rays.
- Stretching (symmetric or asymmetric) involves the change in bond lengths and bending (scissoring, rocking, wagging, and twisting) involves the change in bond angles. The most useful bands in an infrared spectrum correspond to stretching frequencies.
- Compounds (organic or inorganic) having covalent bonds absorb electromagnetic radiations in the infrared region. The main use of the technique is to determine the functional groups in the molecule.
- Our eyes also serve as a visible spectrometer for identifying different substances.
- White light is made up of different colour radiations.
- The molecular formula of a substance is determined by the use of nuclear magnetic resonance (NMR) spectroscopy effectively.

Multiple Choice Questions

Four answers are given for each question. Select the correct answer.

i) Volumetric analysis method is aimed for:
(a) Analyzing solutions (b) Analyzing liquids only
(c) Analyzing solids only (d) Analyzing volume only

ii) The combustion analysis methods are:
(a) Only limited to organic compounds
(b) Only limited to Inorganic compounds
(c) Covered both organic and inorganic compounds
(d) Covered only physical characters of a chemical compound.

iii) The salt analysis covers:
(a) Analysis of organic compounds
(b) Analysis of Inorganic compounds
(c) Both organic and inorganic compounds
(d) Analysis the sodium chloride only.

iv) Mass spectrometers separates ions on which of the following:
(a) Mass (b) Charge
(c) Mass-to-charge ratio (d) Molecular mass

v) The Ultraviolet light has energy:
(a) More than red light (b) Less than red light
(c) More than X-rays (d) More than gamma rays

vi) The red light has wave length from:
(a) 400-450nm (b) 450-550nm
(c) 550-620nm (d) 620-800nm

vii) The portion of the fire that feels us warm is
(a) Ultraviolet rays (b) Infrared rays
(c) Visible radiations (d) Yellow light of fire

viii) The globar rod in IR spectrometer is made of:
(a) Oxides of zirconium (b) Silver carbide
(c) Oxides of cerium (d) Silicon carbide

ix) The colour of a chemical compound results from
(a) Stretching of molecules (b) Bending of molecules
(c) Release of energy (d) Electronic transitions

x) Mass spectroscopy is used to determine
(a) Molecular masses (b) Functional group
(c) Type of atoms (d) Unsaturation

Short Answer Questions

Q.1. Define chemical analysis. What are the main types of chemical analysis?

Q.2. What is the importance of qualitative analysis and quantitative analyses?

Q.3. What are the different types of spectroscopy?

Q.4. What is the main use of IR spectroscopy?

Q.5. Why most chemist use wave number unit rather than wavelength in IR spectroscopy?

Q.6. How UV-Vis spectroscopy differs from IR spectroscopy?

Q.7. How does the detector in NMR spectrometer work?

Q.8. What are the principles of mass spectrometer?

Q.9. Name the factors that affect vibrational frequency.

Q.10 Give three applications of mass spectroscopy.

Long Answer Questions

Q.1. What is chemical analysis? Explain qualitative and quantitative analysis with at least one example.

Q.2. What do you know about classical and modern methods? Compare classical methods of analysis with modern methods.

Q.3. What is the difference between volumetric and gravimetric methods of analysis?

Q.4. What is combustion analysis? Discuss the procedure of combustion analysis.

Q.5. What are electromagnetic radiations? Describe the regions in electromagnetic spectrum in detail?

Q.6. Define and explain atomic absorption and emission spectra.

Q.7. Define spectroscopy. What are the applications of spectroscopy?

Q.8. What is UV-Vis spectroscopy? Explain the principle and applications of UV-Vis spectroscopy.

Q.9. Explain the various electronic transitions possible on the absorption of UV-Visible radiation.

Q.10. Write a note on infrared spectroscopy? Describe the principle and applications of IR spectroscopy.

Q.11. Explain the various vibrations possible when a molecule absorbs infrared radiation.

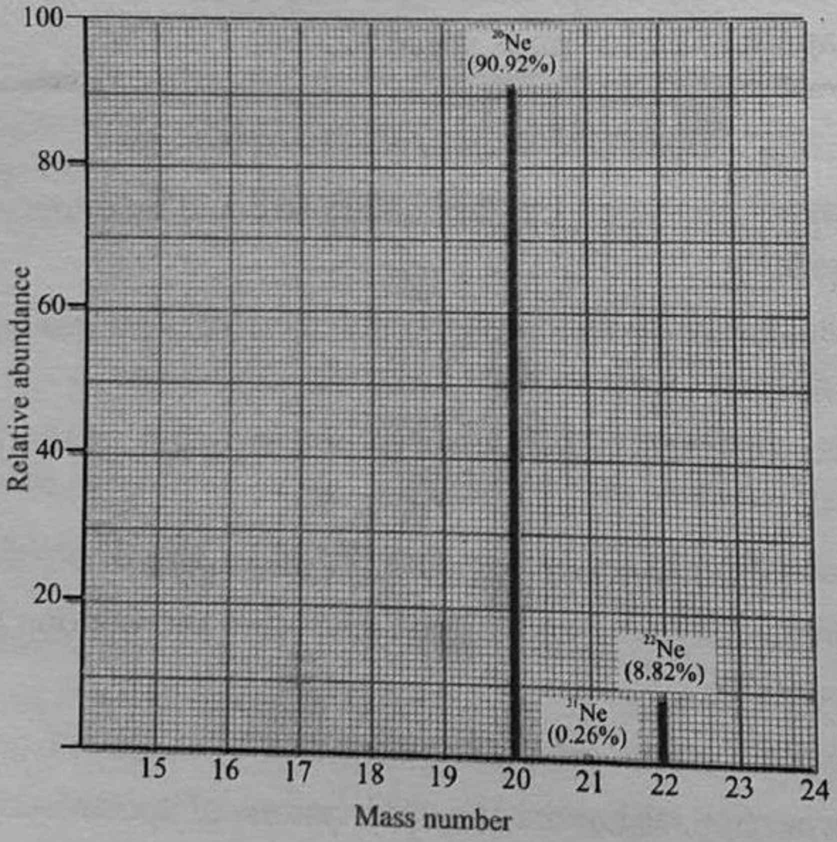
Problems

Q.1. When 2.203g of sample of an organic compound was burnt in a combustion tube, 4.401g of CO_2 and 1.802g of H_2O were produced. Determine the % age of carbon, hydrogen and oxygen atoms.

Q.2. An unknown compound contains 14.31% sodium, 9.97% sulphur, 6.22% hydrogen and 69.5% oxygen. Calculate the empirical formula of the compound.

Q.3. The empirical formula of glucose is CH_2O. It molecular mass is 180 amu. Determine it molecular formula.

Q.4. Calculate the average atomic mass of neon with the help of mass spectrum given below:



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Glossary

Acetals. Acetals are formed when hemiacetals react with second molecule of alcohol.

Achiral: A molecule that is superimposable on its mirror image. It has a lack of handedness.

Activating group: The electron donating groups that increase the reactivity of the aromatic ring towards electrophilic substitution such as hydroxyl group (—OH).

Acylation: The replacement of hydrogen of benzene by an acyl group (—COR) is called alkylation.

Addition Reactions: The most important reactions of alkenes are the addition reactions.

Alcohols: Alcohols are organic compounds in which hydroxyl group is attached to alkyl group.

Alkanes: Alkanes are saturated hydrocarbons containing single bonds between carbon atoms.

Alkenes: Unsaturated hydrocarbons containing one or more carbon-carbon double bonds in their structures.

Alkylation: The replacement of hydrogen of benzene by an alkyl group (—R) is called alkylation.

Alkyl Group: Alkyl group is formed by the removal of one hydrogen atom from an alkane.

Alkyl Halides: Organic compounds in which one or more hydrogen atom(s) of an alkane are replaced by one or more halogen atom(s).

Alkynes: Unsaturated hydrocarbons containing one or more carbon-carbon triple bonds in their structures.

Amines: Organic compounds in which one or more hydrogen atom(s) of ammonia are replaced by alkyl or aryl groups.

Ammonolysis: The process of cleavage of the carbon-halogen bond by ammonia molecule.

Anomers: Similar molecules differing in the configuration at the hemiacetal (anomeric) carbon (carbon–1 atom of an aldose or the carbon–2 atom of a 2–ketose).

Apoenzyme: The proteinaceous part of enzyme.

Bond angle: The angle formed between two adjacent bonds.

Bond Enthalpy: Bond enthalpy, also known as bond energy, is the amount of energy required to break one mole of covalent bonds in a gaseous substance.

Carbocation: Carbocations are carbon atoms in an organic molecule bearing positive charge.

Carbohydrate: Polyhydroxyaldehyde or polyhydroxyketones or compounds which give such compounds on hydrolysis. They include sugars, starches, celluloses, and gums and serves as a major energy source in the diet of animals.

Carbolic Acid: The 5% aqueous solution of phenol.

Carbonyl Group: A functional group composed of a carbon atom doubly bonded to an oxygen atom ($C{=}O$)

Catenation: The self-linking property of atoms of an element to form chains and rings.

Catalytic Hydrogenation: The addition of hydrogen across a double or triple bond of an unsaturated hydrocarbon in the presence of catalyst.

Chiral: A molecule that is not super imposable on its mirror image. It has handedness. The objects that show handedness are golf clubs, scissors, shoes and a corkscrew.

Chiral carbon atom: A carbon atom that is bonded to four different atoms or groups.

Chiral centers: Tetrahedral atoms usually carbon atoms having four different substituents.

Cis-trans Isomers: Compounds having same molecular and structural formulas but restricted rotation about certain bonds.

Coenzyme: The non- proteinaceous part of the enzyme.

Complex Ion: An ion in which a central metal atom or ion is surrounded by ligands (group of ions or molecules).

Deactivating group: The electron withdrawing groups that decrease the reactivity of the aromatic ring towards electrophilic substitution such as nitro group ($—NO_2$).

Decarboxylation: The loss of carbon dioxide from the carboxyl group of a molecule.

Dehydration: A chemical reaction that involves the loss of a water molecule from the reacting molecule. The alcohols can be dehydrated to produce alkenes.

Delocalization: The distribution of electron density over more than two atoms that are bonded together.

Delocalization energy: See resonance energy.

Dextrorotatory: A substance that rotates plane polarized light in a clockwise direction.

Diamagnetism: A form of magnetism whereby certain materials are slightly repelled by an external magnetic field.

Electrophile: Positively charged or neutral specie that can accept an electron pair in a reaction.

Elimination Reaction: A chemical reaction that involves the removal of two atoms or group of atoms from two adjacent carbon atoms of a substrate molecule.

Electromagnetic Spectrum: The entire range of wavelengths or frequencies of electromagnetic radiation extending from the highest energy gamma rays to the lowest energy radio waves.

Empirical Formula: The simplest formula that indicates the relative proportion of atoms present in a compound. It does not show actual number or arrangement of atoms.

Enzymes: Biological molecules that act as catalysts within living cells.

Epoxidation: The addition of oxygen (O_2) to alkene is called epoxidation.

Esterification: An equilibrium reaction in which an alcohol and an acid (carboxylic acid) form an ester as the reaction product.

Ethers: A class of organic compounds that consist of an oxygen atom bonded to two alkyl or/and aryl groups.

Fat: Triesters of glycerol and fatty acids. They are generally soluble in organic solvents and largely insoluble in water.

Fibres: Thin threads of natural or artificial substances from which a vegetable tissue, mineral substance, or textile is formed. Natural fibres include cotton, hair, fur, silk, and wool. Natural fibres are of plant or animal origin. The most important synthetic fibres are dacron, nylon, orlon, and polypropylene.

Fatty Acids: A carboxylic acid with a long aliphatic chain, which is either saturated or unsaturated.

Fullerenes: An allotrope of carbon in the form of a hollow sphere, ellipsoid, tube, and many other shapes.

Functional Group: An atom or group of atoms within a molecule which confers specific properties to a compound. It is the reactive part of the molecule. Each functional group has its characteristic reactivity.

Grignard Reagents: Alkyl magnesium halides (RMgX) are commonly known as Grignard Reagents.

Groups: Columns of elements in the periodic table.

Halohydration: The addition of hypohalous acid to alkenes.

Heat of Hydrogenation: The amount of heat evolved when one mole of an alkene is hydrogenated.

Hemiacetals: These compounds are formed by the addition of an alcohol molecule to carbonyl group. On the other hand, acetals are formed when hemiacetals react with second molecule of alcohol.

Homologous Series: A series of compounds with the same functional group in which each member differs from the next member by a methylene (—CH_2—) group in their formula.

Hydration: The addition of water to an unsaturated compound particularly an alkene or alkyne.

Hydrohalogenation: The addition of HX to alkenes.

Hydrocarbons: Compounds containing atoms of carbon and hydrogen only.

Hydrogenation: The addition of hydrogen across a double or triple bond of an unsaturated hydrocarbon.

Isomers: Compounds having same molecular formula but different structures.

Laevorotatory: A substance that rotates plane polarized light in an anticlockwise direction.

Lipids: They include fatty acids, neutral fats, waxes and steroids (like cortisone). They are soluble in organic solvents such as alcohol, ether and chloroform. They are insoluble in water.

Markovnikov's Rule: According to this rule, the negative part of the polar reagent (unsymmetrical molecule) like HX adds to that carbon of double bond of an unsymmetrical alkene which has minimum number of hydrogen(s).

Mercapto Group: A functional group containing a sulphur atom bonded to a hydrogen atom. Its general formula is —SH. It is also known as thiol group or sulfhydryl group.

Monomers: They are the building blocks from which polymers are made.

Ozonolysis: The reaction of ozone (O_3) with alkene to break carbon-carbon double bond.

Peptides: They are short chain of amino acids monomers linked by peptide bonds.

Period: Rows of elements in the periodic table.

Periodic Table: A table of elements in which elements are arranged into groups and periods in the increasing order of atomic numbers.

Phosphodiester Bond: A covalent bond in RNA or DNA that holds a polynucleotide chain together by joining a phosphate group at position 5 in the pentose sugar of one nucleotide to the hydroxyl group at position 3 in the pentose sugar of the next nucleotide.

Phosphodiester Linkage: See phosphodiester bond.

Polarimeter: An instrument used to measure the optical activity of a compound.

Polymerization: The chemical process of formation of large molecule by joining together of two or more small molecules by chemical bonds.

Proteins: They are composed of long chains of amino acids. They are essential part of all living organisms.

Resonance Energy: The resonance energy of a compound is a difference between the energy of the actual molecule and the most stable resonance structure. It is also known as delocalization energy.

Resonance: The process in which two or more structures are written for a compound which differ only in the arrangement of electrons is called resonance.

Saturated Hydrocarbons: Hydrocarbons with only single covalent bonds.

Shelf Life: The period of time that a product can be stored and remain fresh, useful or suitable for consumption.

Steric Hindrance: The prevention or retardation of chemical reaction because of the blocking of the reactive site of a molecule by adjacent atoms or groups of atoms.

Stereoisomerism: Compounds that have same connectivity but different arrangement of atoms or group of atoms in space are called stereoisomers and the phenomenon is known as stereoisomerism.

Spectroscopy: The study of the interaction between matter and electromagnetic radiation is called spectroscopy.

Substrate: A substrate is a molecule undergoing a reaction. It may also be defined as: the molecule that is attacked in a chemical reaction.

Sulfhydryl Group: See mercapto group.

Tautomerism: The isomers of a compound which differ only in the position of protons and electrons are called tautomers and the phenomenon is known as tautomerism.

Unsaturated Hydrocarbons: Hydrocarbons with multiple (double or triple) covalent bonds.