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Review Article

A CONCISE REVIEW: ON VARIOUS ANALYTICAL INSTRUMENTS

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ABSTRACT

Analytical Instrument work by different way such as production of signal of sample, absorption of radiation, Emission of radiation, scattering of radiation, refraction of radiation. Etc. The resulting signal may be used directly or transformed to one of different nature. The correlation between blank reading / signal & sample signal can be understood only if the instruments are understood properly. For example in U.V. Spectroscopy sample absorb the radiation of certain wavelengths. The degree of absorption is then correlated with the concentration of the particular ion which was in the solution (sample). This review try to focus on General Introduction, principle, and Instrumentation of various Instrument like U.V, IR, N.M.R, atomic absorption spectroscopy, Coulometry, Flame photometer Raman Spectroscopy, High Performance Liquid Chromatography, gas chromatography.

INTRODUCTION

Analytical chemistry defined as the science and art of determining the composition of materials in terms of the elements or compounds contained in them. Analytical chemistry is a measurement science consisting of a set of powerful ideas and methods that are useful in all fields of science and medicine. The ability to provide timely, accurate and reliable data is central to the role of analytical chemists and is especially true in the discovery, development and manufacture of pharmaceuticals. Thus the most manufacturing industries rely upon both qualitative and quantitative chemical analysis to ensure that raw material used meet certain specifications and also to check the quality of the final product for this purpose analytical chemist rely upon various analytical instrument like Optical (Refractometry, Polarimetry, Emission Spectrophotometry and Nephelometry or Turbidometry), Electrochemical (Potentiometry, Amperometry and Polarography) and Chromatography (Paper, Column, Thin Layer, Gas Liquid Chromatography, High Performance Liquid Chromatography by using this we can check various Physicochemical property. Methods involving nuclear reaction like Nuclear Magnetic Resonance happened to be more popular. GC-MS combination or hyphenated system is one of the prominent powerful tools available for drug analysis. The chemical methods include the volumetric and gravimetric procedures, which are mainly, depend on complex formation, the modern methods (HPLC, UPLC, GLC, GC-MS/MS, LC-NMR and Liquid chromatography-mass spectrometry are the available choices for assay involving sophisticated equipment, which are highly sensitive, accurate and consume very negligible amount of samples for analysis.

In general we can say that an analytical instrument does not produce quantitative data rather it converts chemical information to a form more readily observable form.

That means instrument act as medium of communication device. The Table no 1 contains various instruments and their principle.

Spectroscopy: When a molecule is exposed to an electromagnetic radiation certain amount of energy associated with the particular radiation is absorbed by molecule. As a molecule absorbs energy, an electron is promoted from an occupied orbital to an unoccupied orbital of greater potential energy. Generally, the most probable transition is from the highest occupied molecular orbital to the lowest unoccupied molecular orbital.

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Table no1 List of instrument

Name of Instrument	Principle
U.V Visible Spectrophotometer, I.R. Spectrophotometer. Coulmetry, Atomic absorption Spectrophotometer. N.M.R. Spectrophotometer.	Absorption Of Radiation
X-RAY, Flame Photometry, Fluorescene photometry.	Emission of Radiation
Turbidometry, Nephelometry, Raman spectroscopy.	Scattering of radiation
Refractometry	Refraction of radiation
Electron diffraction method	Diffraction of radiation
Polarimetry, optical rotatory dispersion	Rotation of radiation
Potentiometry	Electrical potential
Conductivity	Electrical conductance
Mass spectroscopy	Mass- to charge Ratio
Thermal conductivity	Thermal Properties
HPLC	Liquid- liquid speration
Gas Chromatography	Gas –Liquid , Gas-Solid separtion

The energy differences between electronic levels in most molecules vary from 125 to 650 KJ/mole.[4] absorption of light in UV and visible regions gives rise to the absorption spectra. An absorption spectrum is observed, when radiation of range of frequencies is passed through a substance. The range of radiation in EMR spectrums [4] used in absorbance spectrum is given in Table 2.

Table no. 2 Range of Radiation in EMR Spectrum

Region of spectrum	Wavelength range
Far UV	100-200 nm
Near UV	200-400 nm
Visible	400-780 nm
Near IR	0.78-4 µm
IR	4-25 µm

Molecular spectra are considerably more complex, because molecules are capable of absorbing energy in several ways; there are three basic internal energy levels in molecules. The electronic transitions involved in the UV-visible region are as follows. $n \rightarrow \pi^*$, $n \rightarrow \sigma^*$, $\pi \rightarrow \pi^*$, $\sigma \rightarrow \pi^*$, $\sigma \rightarrow \sigma^*$. The energy required for the $\sigma \rightarrow \sigma^*$ transition is very high; consequently compounds in which all valence shell electrons are involved in single bond formation, such as saturated hydrocarbon do not absorb in ordinary UV region. An exception is cyclopropane, which shows λ-max at about 190 nm. From the molecular orbital diagram (Fig-1), there are several possible electronic transitions that can occur, each of a different relative energy.

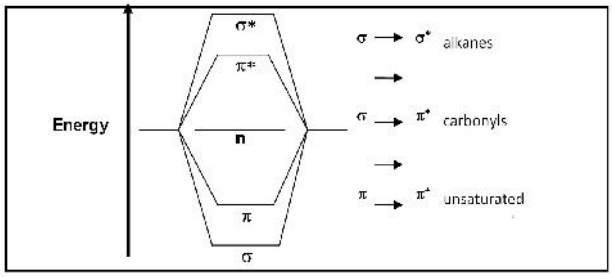


Figure. no. 1 Electronic Energy Levels and Transitions

The high energy transition ($\sigma \rightarrow \sigma^*$) occurs at shorter wavelength and low energy transitions ($\pi \rightarrow \pi^*$) occur at longer wavelength. Excited molecules returned to the ground state within 10-8 sec.

UV– Visible Spectrophotometry

Principle: The name UV means "beyond violet" (from Latin ultra, "beyond"), violet being the color of the shortest wavelengths of visible light. The color violet has the shortest wavelength in the visible spectrum. UV light has a shorter wavelength than that of violet light. One among the various analytical techniques used for analysis of mixtures is spectrophotometry. It involves separation of drugs by physical and chemical methods and then to read them at their λ max to obtain results. This technique involves two steps and has relatively low sensitivity. Derivative spectroscopy is slight advanced which do not require separation but the difference in λ max of component of drugs need to be at least 20 nm. [5]

Insturment: Typical spectrophotometer possesses the following components.

A Source of radiation*:* It is important that the power of the radiation source does not change abruptly over its wavelength range. The electrical excitation of deuterium or hydrogen at low pressure produces a continuous UV spectrum. The mechanism for this involves formation of an excited molecular species, which breaks up to give two atomic species and an ultraviolet photon both Deuterium and Hydrogen lamps emit radiation in the range 160 - 375 nm. Quartz windows must be used in these lamps, and quartz cuvettes must be used, because glass absorbs radiation of wavelengths less than 350 nm.

Various UV radiation sources are as follows

1. Deuterium lamp
2. Hydrogen lamp
3. Tungsten lamp
4. Xenon discharge lamp
5. Mercury arc lamp

Various Visible radiation sources are as follows

1. Tungsten lamp
2. Mercury vapour lamp
3. Carbonone lamp

A Monochromator: All monochromators contain the following component parts;

1. An entrance slit
2. A collimating lens
3. Dispersing device (a prism or a grating)
4. A focusing lens
5. An exit slit

Polychromatic radiation (radiation of more than one wavelength) enters the monochromator through the entrance slit. The beam is collimated, and then strikes the dispersing element at an angle. The beam is split into its component wavelengths by the grating or prism. By moving the dispersing element or the exit slit, radiation of only a particular wavelength leaves the Monochromator through the exit slit.

A Sample Holder: A variety of sample cells available for UV region. The choice of sample cell is based on

1. The path length, shape, size
2. The transmission characteristics at the desired wavelength

3. The relative expense

The cell holding the sample should be transparent to the wavelength region to be recorded. Quartz or fused silica cuvettes are required for spectroscopy in the UV region. Silicate glasses can be used for the manufacture of cuvettes for use between 350 and 2000nm. The thickness of the cell is generally 1 cm. cells may be rectangular in shape or cylindrical with flat ends.

A Detector: In order to detect radiation, three types of photosensitive devices are

1. Photovoltaic cells or barrier- layer cell
2. Phototubes
3. Photomultiplier tubes

Photovoltaic cell*:* is also known as barrier layer cell. It consists of a metallic base plate like iron or aluminum which acts as one electrode. On its surface, a thin layer of a semiconductor metal like selenium is deposited. Then the surface of selenium is covered by a very thin layer of silver or gold which acts as a second collector tube.

When the radiation is incident upon the surface of selenium, electrons are generated at the selenium- silver surface and the electrons are collected by the silver. This accumulation at the silver surface creates an electric voltage difference between the silver surface and the basis of the cell.

Phototubes: Are also known as photo-emissive cells. A phototube consists of an evacuated glass bulb. There is light sensitive cathode inside it. The inner surface of cathode is coated with light sensitive layer such as potassium oxide and silver oxide.

When radiation is incident upon a cathode, photoelectrons are emitted. These are collected by an anode. Then these are returned via external circuit. And by this process current is amplified and recorded.

The photomultiplier tube: It is a commonly used detector in UV spectroscopy. It consists of a Photo-emissive cathode (a cathode which emits electrons when struck by photons of radiation), several dynodes (which emit several electrons for each electron striking them) and an anode. A photon of radiation entering the tube strikes the cathode, causing the emission of several electrons. These electrons are accelerated towards the first dynode (which is 90V more positive than the cathode). The electrons strike the first dynode, causing the emission of several electrons for each incident electron. These electrons are then accelerated towards the second dynode, to produce more electrons which are accelerated towards dynode three and so on. Eventually, the electrons are collected at the anode. By this time, each original photon has produced 106 - 107 electrons. The resulting current is amplified and measured. Photomultipliers are very sensitive to UV and visible radiation. They have fast response times. Intense light damages photomultipliers; they are limited to measuring low power radiation.

A Recorder: Amongst the instrumental methods spectrophotometry occupies very important position, which utilizes the measurement of intensity of electromagnetic radiation, emitted or absorbed by the analyte. The careful manipulation of the experimental parameters in UV- visible spectrophotometry often enables the analyte to estimate simultaneously more than one component present in a mixture without their prior separation. Schematic representation of UV double beam spectrophotometry given in (Fig 2).

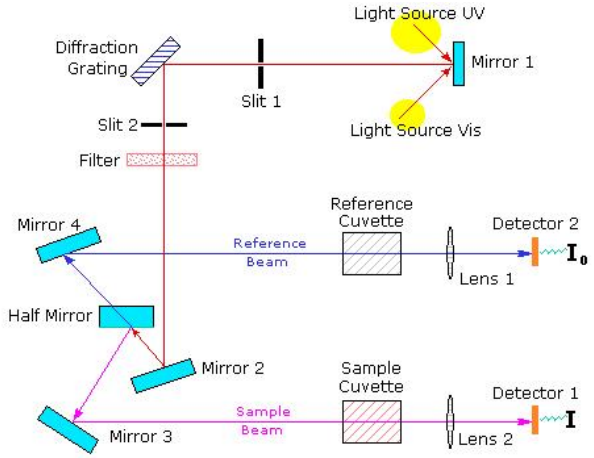
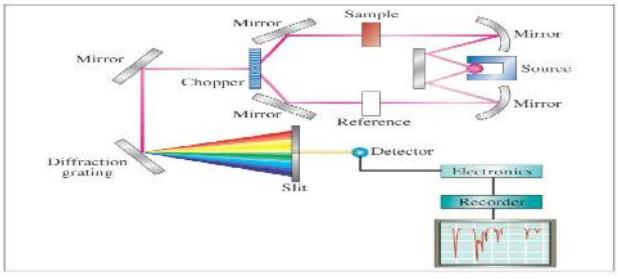


Figure no 2 Schematic diagram of UV Double Beam Spectrophotome

Infra-Red Spectroscopy

Principle: Infrared spectroscopy (IR spectroscopy or vibrational spectroscopy) involves the interaction of infrared radiation with matter. It covers a range of techniques, mostly based on absorption spectroscopy. As with all spectroscopic techniques, it can be used to identify and study chemicals. Samples may be solid, liquid, or gas. The method or technique of infrared spectroscopy is conducted with an instrument called an infrared spectrometer (or spectrophotometer) to produce an infrared spectrum. An IR-spectrum is essentially a graph of infrared light absorbance or transmittance on the vertical axis vs. frequency or wavelength on the horizontal axis. Typical units of frequency used in IR spectra are reciprocal centimeters (sometimes called wave numbers), with the symbol cm^{-1}. Units of IR wavelength are commonly given in micrometers (formerly called "microns"), symbol µm, which are related to wave numbers in a reciprocal way. A common laboratory instrument that uses this technique is a Fourier transform infrared (FTIR) spectrometer. Two-dimensional IR is also possible as discussed below.



Instrument Figure no. 3 Schematic Diagram of Infra Red Spectroscopy.

The infrared portion of the electromagnetic spectrum is usually divided into three regions; the near-, mid- and far- infrared, named for their relation to the visible spectrum. The higher-energy Near-IR, approximately 14000–4000 cm^{-1} (0.8–2.5 µm wavelength) can excite overtone or harmonic vibrations. The mid-infrared, approximately 4000–400 cm^{-1} (2.5–25 µm) may be used to study the fundamental vibrations and associated

rotational structure. The far-infrared, approximately 400–10 cm^{-1} (25–1000 μm), lying adjacent to the microwave region, has low energy and may be used for rotational spectroscopy. The names and classifications of these sub regions are conventions, and are only loosely based on the relative molecular or electromagnetic properties.

Typical Infra Red spectroscopy posses the following components

IR Radiation source: IR instruments require a source of radiant energy which emits IR radiation which must be steady, intense enough for detection and extend over the desired wavelength. Various sources of IR radiations are as follows.

a. Nernst glower
b. Incandescent lamp
c. Mercury arc
d. Tungsten lamp
e. Glober source
f. Nichrome wire

Mono-chromators: Various types of monochromators are prism, gratings and filters. Prisms are made of Potassium bromide, Sodium chloride or Caesium iodide. Filters are made up of Lithium Fluoride and Diffraction gratings are made up of alkali halides.

Sample cells & Sampling of substances: IR spectroscopy has been used for the characterization of solid, liquid or gas samples.

Solid-Various techniques are used for preparing solid samples such as pressed pellet technique, solid run in solution, solid films, mull technique etc.

Liquid-samples can be held using a liquid sample cell made of alkali halides. Aqueous solvents cannot be used as they will dissolve alkali halides. Only organic solvents like chloroform can be used.

Detectors: Detectors are used to measure the intensity of unabsorbed infrared radiation. Detectors like thermocouples, Bolometers, thermisters, Golay cell, and pyro-electric detectors are used.

Coulmetry: Coulometry is the name given to a group of techniques in analytical chemistry that determine the amount of matter transformed during an electrolysis reaction by measuring the amount of electricity (in coulombs) consumed or produced. There are two basic categories of coulometric techniques. Potentiostatic coulometry involves holding the electric potential constant during the reaction using a potentiostat. The other called Coulometric titration or Amperostatic coulometry. Keeps the current (measured in amperes) constant using an amperostat.

Potentiostatic coulometry: It is a technique most commonly referred to as "bulk electrolysis". The working electrode is kept at a constant potential and the current that flows through the circuit is measured. This constant potential is applied long enough to fully reduce or oxidize all of the electro-active species in a given solution. As the electro-active molecules are consumed, the current also decreases, approaching zero when the conversion is complete. The sample mass, molecular mass, number of electrons in the electrode reaction, and number of electrons passed during the experiment are all related by Faraday's laws. It follows that, if three of the values are known, then the fourth can be calculated.

An advantage to this kind of analysis over electrogravimetry is that it does not require that the product of the reaction be weighed. This is useful for reactions where the product does not deposit as a solid, such as the determination of the amount of arsenic in a sample from the electrolysis of arsenous acid (H_3AsO_3) to arsenic acid (H_3AsO_4).

Coulometric titration: Coulometric titrations use a constant current system to accurately quantify the concentration of a species. In this experiment, the applied current is equivalent to a titrant. Current is applied to the unknown solution until all of the unknown species is either oxidized or reduced to a new state, at which point the potential of the working electrode shifts dramatically. This potential shift indicates the endpoint. The magnitude of the current (in amperes) and the duration of the current (seconds) can be used to determine the moles of the unknown species in solution. When the volume of the solution is known, then the molarity of the unknown species can be determined.

Instrumentation

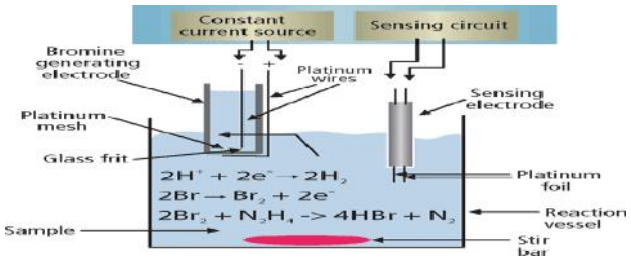


Figure no. 4 Schematic diagram of Coulometry

Nuclear Magnetic Resonance

Principle: The principle of nuclear magnetic resonance is based on the spins of atomic nuclei. The magnetic measurements depend upon the spin of unpaired electron whereas nuclear magnetic resonance measures magnetic effect caused by the spin of protons and neutrons. Both these nucleons have intrinsic angular momenta or spins and hence act as elementary magnet. The existence of nuclear magnetism was revealed in the hyper fine structure of spectral lines. If the nucleus with a certain magnetic moment is placed in the magnetic field, we can observe the phenomenon of space quantization and for each allowed direction there will be a slightly different energy level. The nuclei of all elements carry a charge. When the spins of the protons and neutrons comprising these nuclei are not paired, the overall spin of the charged nucleus generates a magnetic dipole along the spin axis, and the intrinsic magnitude of this dipole is a fundamental nuclear property called the nuclear magnetic moment, μ. Nuclei that exhibit the NMR phenomenon are those which have the spin quantum number I greater than 0 (I>0). A nucleus with an odd mass or an odd atomic number possess a nuclear spin, due to spinning a magnetic field is generated along the axis. The spin quantum number I of the nuclei as follows: Without externally applied magnetic field, the nuclear spins are random in all directions. But when externally magnetic field is applied; the nucleus align themselves by creating magnetic momentum.

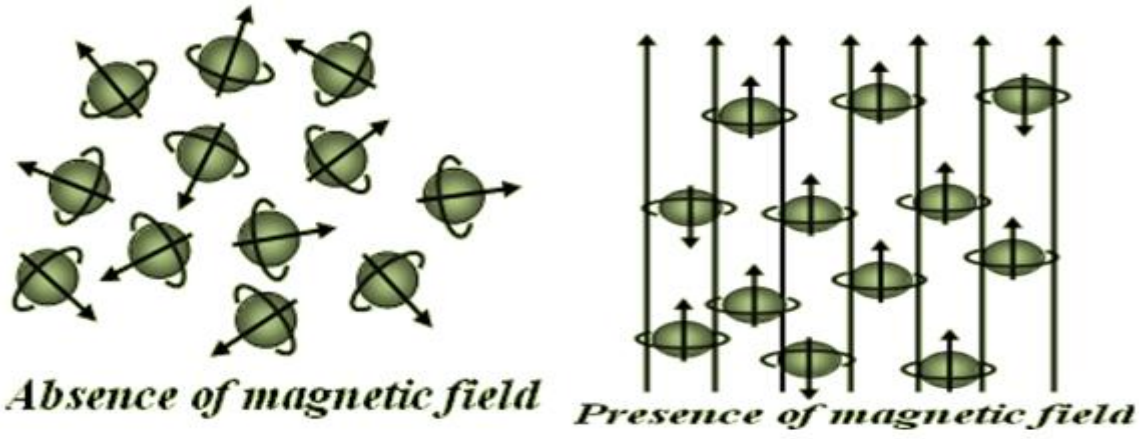


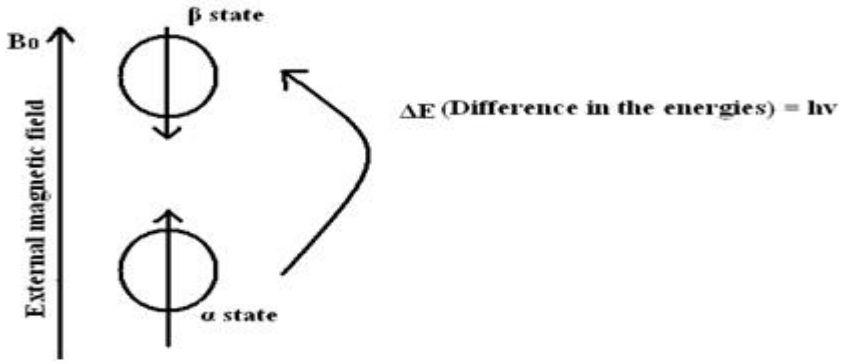
Figure 5 Orientation of spinning nuclei in absence and presence of external magnetic field

Hence, nucleus spins on their own axis when placed in an external magnetic field resulting in a circular motion creating a precessional orbit, with a frequency called Precessional frequency. When energy in the form of radio frequency is applied and is equal to precessional frequency, then the transition of protons from lower energy (α state) to higher energy (β state) take places and NMR signals are recorded.

Instrumentaion

Sample Holder

Usually the dimension of sample holder is 8.5cm in length and 0.3 mm diameter. Glass tubes are generally used as sample holder as these are more economic. The following ideal characteristics are there in the sample holder.



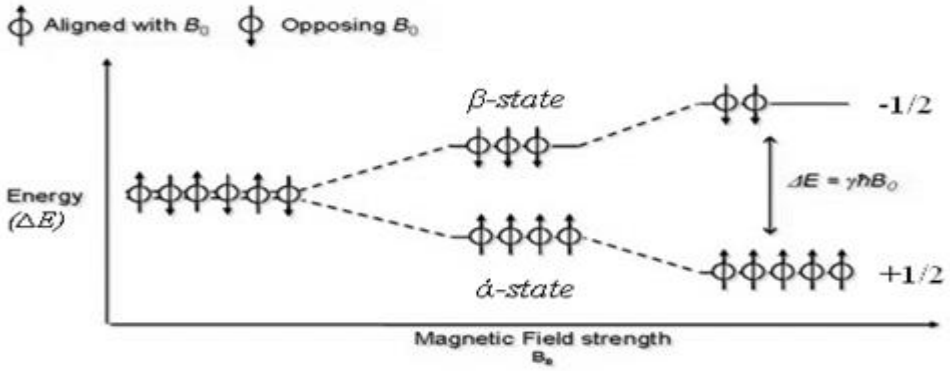


Figure 6 Energy level transitions of protons

When application of radio frequency energy is stopped nucleus returns to ground state. Increasing in strength of magnetic field does not cause transition from lower energy (α state) to higher energy (β state). But it merely increases precessional frequency.

- It should be sturdy.
- It should be practical.
- It should be cheap.
- It should be transparent to radio frequency radiations.
- It should be chemically inert.

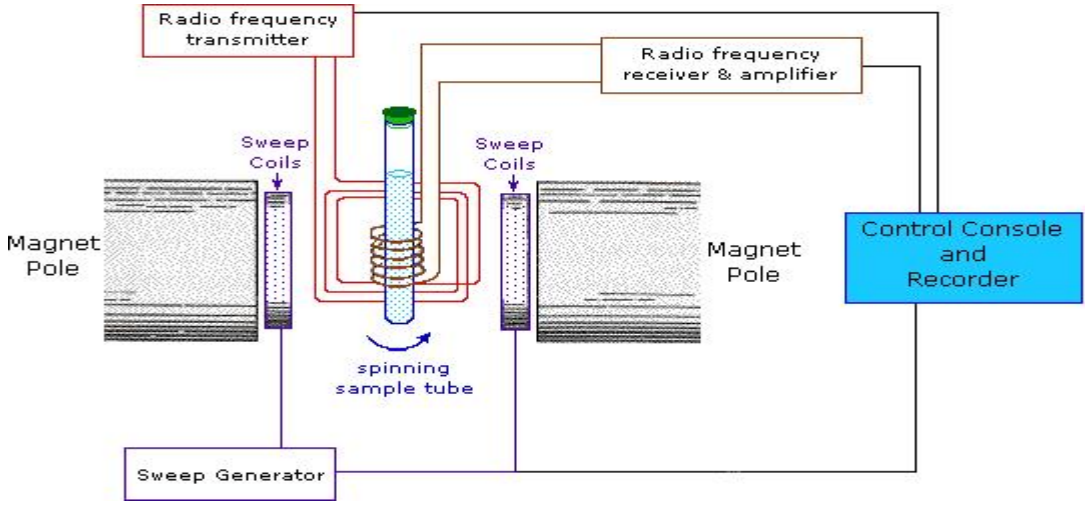


Figure no 7 Schematic diagram of nuclear magnetic resonance

Sample probe

It holds the sample tube in a magnetic field and rotates it along its axis, resulting in sharper lines with better resolution due to decrease in the effects of in homogeneities in the magnetic field. The probe may be either a single coil or system of coils depending upon the type of instrument.

Permanent Magnet

Permanent magnet or electromagnet has the important feature that it should give homogeneous magnetic field, i.e., the strength and direction of the magnetic field should not change from point to point. As the field strength is proportional to the chemical shifts, it must not be less than 20,000\ guass.

Magnetic Coils

It is employed for the production of NMR spectra. It is achieved by passing direct current either through the coils that are wound around the magnetic pole or through a pair of Helmholtz coils located on either side of the sample probe. The relationship between the resonance frequency of the nucleus and the strength of magnetic field (H0) is expressed as:

From that equation, frequency is directly proportional to strength of magnetic field (H0). If H0 is kept constant, the precession frequency is fixed. If radiofrequency is kept constant, the resonance frequency of the nucleus must be changed by varying H0.

Sweep Generator

If precession frequency is equal to applied frequency radiations, this results in nucleus to resonate. Sweep generator method, is used to vary the magnetic field and it is easier, than the variation of radio frequency.

Radio Frequency Generator

Radio Frequency Generator is also known as Radio Frequency Transmitter. In order to generate radio frequency radiation, radio frequency oscillator is used which irradiates the sample molecules. Due to applied radiofrequency, an energy difference occurs and the nuclei moves from ground state to excited state. The coil surrounding the sample results in resonance signals.

Radio Frequency Receiver: When the radio frequency radiation is passed through the magnetized sample two phenomena namely absorption and dispersion may occur. The observation of either absorption or dispersion will enable the resonance frequency to be determined.

For the Detection of Resonance Signal following Methods are used

Radio Frequency Bridge: is employed under single coil instruments. It allows absorption and dispersion signals to appear as an output of EMF across the bridge. Signals can be recorded mechanically.

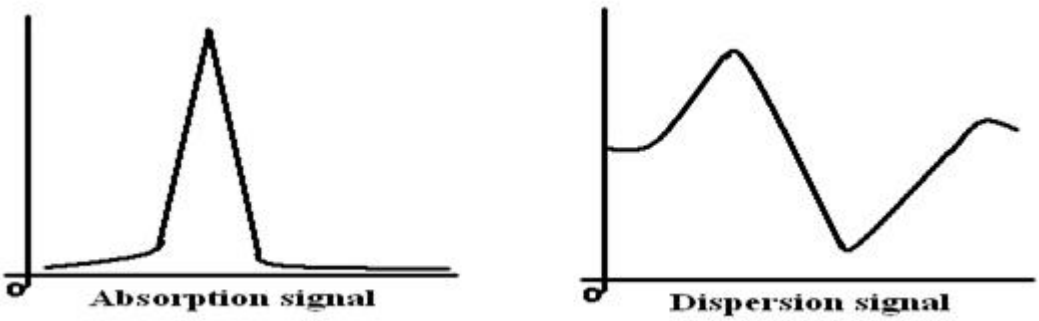


Fig 5 Absorption and Dispersion signals

Crossed coil or Nuclear induction method: This method employs a separate receiver coil in this the transmitter and receiver coils are arranged perpendicular to each other and to the direction of the magnetic field.

Amplifier

The absorption signal received from radio frequency receiver is extremely weak. Therefore, it requires considerable amplification before it is fed to a chart recorder in which amplifier is used for amplification of weak signals.

Read Out

The NMR spectra obtained from instruments are directly recorded via a computer or even mechanically.

Flame Photometry

Principle: When a solution of inorganic salt is sprayed as fine droplets into a flame. Due to heat of the flame, the droplets dry leaving a fine residue of salt. This fine residue converts into neutral atoms. Due to the thermal energy of the flame, the atoms get excited and there after return to ground state mean while the ions of the metal absorb the electrons from the flame and get reduced to the elemental state due to high temperature of the flame these atoms are in the vapor form. In case of inefficient atomization the process of dissociation of a molecule into its constituent ions atoms remains incomplete this results in the mixture of vapor of atoms, ions and molecules the flame. The atoms formed after atomization of the sample may absorb energy of the flame or any other excitation source can excite electrons of an atomic sample. The intensity of emission, absorption or fluorescence can be measured for quantitative analysis.

Table no 3 shows Principle at Glance

Liquid sample containing element aspirated into flame.
Formation of the liquid droplets.
Evaporation of droplets resulting in the formation of residue.
Decomposition of residue into neutral atoms.
Formation of excited atoms and emission of radiation from atoms.
Wavelength and Intensity of emitted radiation measured by flame photometry.

Insturment: Typical Flame Photometer posses the following components shown in (fig no.8)

1. Burner
2. Mono-chromators
3. Detectors
4. Recorder and display

Burner*:* This is a part which produces excited atoms. Here the sample solution is sprayed into fuel and oxidant combination. A homogenous flame of stable intensity is produced. There are different types of burners like Total consumption burner, Laminar flow and Mecker burner.

Fuel and oxidants*:* Fuel and oxidant are required to produce flame such that the sample converts to neutral atoms and get excited by heat energy.

The temperature of flame should be stable and also ideal. If the temperature is high, the elements in sample convert into ions instead of neutral atoms. If it is too low, atoms may not go to exited state. So a combination of fuel and oxidants is used such that there is desired temperature.

The following combination of fuel and oxidants are commonly used shown in table no 4.

Table.no.4 Commonly used fuel and oxidents

Fuel	Temperature of Flame
Propane +	2100 Degree C
Propane +	2800 Degree C
Hydrogen +	1900 Degree C
Hydrogen +	2800 Degree C
Acetylene +	2200 Degree C
Acetylene +	3000 Degree C

Monochromators

Filters and monochromators are needed to isolate the light of specific wavelength from remaining light of the flame. For this simple filters are sufficient as we study only few elements like Ca, Na, K and Li. So a filter wheel with filter for each element is taken. When a particular element is analyzed, the particular filter is used so that it filters all other wavelengths.

Detector*:* Flame photometric detector is similar to that used in spectrophotometry. The emitted radiation is in the visible region i.e. 400nm to 700nm. Further the radiation is specific for each element so simple detectors are sufficient for the purpose like photo voltaic cells, photo tubes etc.

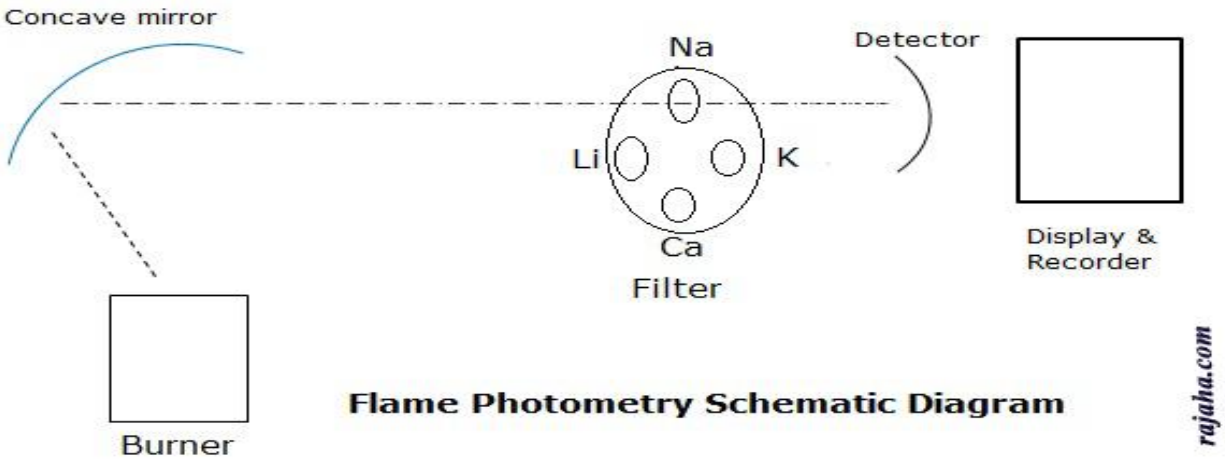


Figure 8 Flame photometry schematic diagram

Recorders and display*:* These are the devices to read out the recording from detectors.

Raman Spectroscopy*:* when substance is irradiated with a monochromatic light of definite frequency the light scattered at right angles to the incident light contains lines of incident frequency, also of lower frequency. Sometimes lines of higher frequency are also obtained thus certain discrete frequencies above and below that of the incident beam will be scattered. It is called raman spectroscopy.

Principle*:* When beam of monochromatic light is passed through liquid or gas a small fraction of it is scattered due to collision between molecules of the scattered and photons of light. When spectroscopy was used to investigate the scattered light .it was found that frequency of the scattered light was same as the frequency of the incident radiations. This phenomenon was observed by Rayleigh and is known as Rayleigh scattering. In 1928 Sir C.V.Raman discovered that when a beam of monochromatic light was allowed to pass though the substance in the solid, liquid or gaseous state, the scattered light contains some additional frequencies over and above that of incident frequency. This is known as Raman Effect. The lines whose wavelength has been modified in Raman Effect are called Raman Lines. The lines having wavelengths greater than that of the incident wavelength are called stoke's lines and those having shorter wavelength anti stoke'slines. If vi is the frequency of incident radiation and vs. the radiation scattered by the given molecular species then the Raman shift delta vi is define by the following relation α= v= vi- vs.

The Raman shift does not depend upon the frequency of the incident light but it is regarded as a characteristic of the substance causing Raman Effect. For stoke lines delta v positive and for antistoke lines delta v is negative.

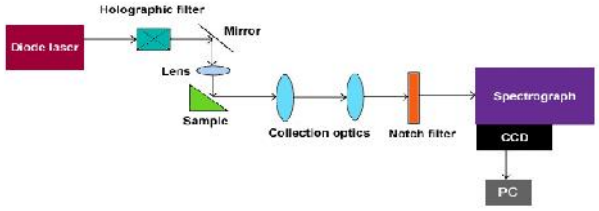


Figure.no 9 Typical Raman Spectrometer

Instrumentation: A Raman spectrometer consist of following three main components

Diode Laser*:* The sampling interface, and the spectrometer itself. A typical Raman laser will consists of different characteristics, such as a small form factor, low power consumption, narrow linewidth, a stable power output, and a stable wavelength output. In case Raman measurements are conducted using a 785nm source, it is important to ensure that the source is only emitting 785nm.

Sampling Interface*.* In many Raman spectrometers, fiber-optic probe are typically used which offers an extremely flexible sampling interface. These fiber-optic probes can be easily adapted to a range of optical microscopes, gas flow cells, liquid flow cells, and other sampling chambers. One critical aspect of a fiber-optic probe is a high-optical-density Raman cutoff. This means, when users are looking at the Raman spectrum, they need to ensure that the laser wavelength is blocked as much as possible so that the Raman shift can be observed. It is extremely important that the Raman shift is observed very close to the laser line since many materials have vital spectral features very near to the line.

Detector*:* Here, important performance factors are small form factor, high resolution, low power consumption, and low noise. An appropriate detector is very important and must be utilized depending on which excitation laser is being used. For visible excitation, a standard CCD is selected; for UV excitation, a photomultiplier tube (PMT) or CCD is typically chosen; and for NIR excitation, an indium gallium arsenide (InGaAs) array is normally employed.

Atomic Absorption Spectroscopy: Atomic absorption spectroscopy is a method of elemental analysis. it is particular useful for determining trace metals in liquid and is almost independent of the molecular form of the metal in the sample.

Principle: when solution containing metallic species is introduced into a flame the vapor of metallic species will be obtained some of the metal atoms may be raised to an energy level sufficiently high to emit the characteristic radiation of thea phenomenon that is utilized in the familiar technique of emission flame photometry. But large percentage of the atoms will remain in the non –emitting ground state these ground state atoms of a particular element are receptive of light radiation of their own specific resonance wavelength thus when alight of this wavelength is allowed to pass through a flame having of the metallic species part of that light will be absorbed and the absorption will be proportional to the density of the atoms in the flame thus in atomic absorption spectroscopy one determine the amount of light absorbed once the value is known the concentration of the metallic element can be known because the absorption is proportional to the density of the atoms in the flame.

Instrument

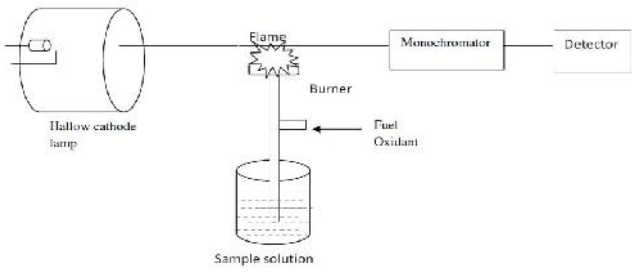


Figure no 10 Typical Atomic absorption spectroscopy

Radiation source: The radiation source for atomic absorption spectrophotometer should emit the stable, intense radiation of the element to be determined this is primary requirement in the entire process. If the element to be analyzed is magnesium, the cathode lamp made of magnesium is used and so for all the other metal elements analyzed like Na, Ca, K, Zn etc.

Chopper: A Rotating wheel is interposed between the hallow cathode lamp and the flame this is rotating wheel is known as chopper and is interposed to break the steady light from the lamp into an intermittent light this gives pulsating current in the

photocell. This pulsating current s amplified and recorded and thus absorption of light will be measured without interference from the light emitted by flame itself.

Atomizer: In order to achieve absorption of atoms it becomes necessary to reduce the sample of the atomic state this done by 1] flame atomizers 2] non –flame atomizers

Nebulization of liquid sample: Formation of the small droplets from the liquid sample is called nebulization A common method nebulisation is by use of a gas moving at high velocity called pneumatic nebulisation

Monochromators: In atomic absorption measurements the most common monochromators are prism and gratings. The function of monochromator is to select a given absorbing line from spectral lines emitted from hallow cathode.

Detectors. The used can be a simple photo multiplier tube or photo cell. The current or potential recorded for the sample absorption is recorded in computer software and then analyzed.

High Performance Liquid Chromatography

Principle

High performance liquid chromatography (HPLC) is basically a highly improved form of column liquid chromatography. Instead of a solvent being allowed to drip through a column under gravity, it is forced through under high pressures of up to 400 atmospheres. That makes it much faster. All chromatographic separations, including HPLC operate under the same basic principle; separation of a sample into its constituent parts because of the difference in the relative affinities of different molecules for the mobile phase and the stationary phase used in the separation.

whose respective concentrations are varied depending on the composition of the sample.

Pump: A pump aspirates the mobile phase from the solvent resorvoir and forces it through the system's column and detecter. Depending on a number of factors including column dimensions, particle size of the stationary phase, the flow rate and composition of the mobile phase, operating pressures of up to 42000 kPa (about 6000 psi) can be generated.

Sample Injector: The injector can be a single injection or an automated injection system. An injector for an HPLC system should provide injection of the liquid sample within the range of 0.1-100 mL of volume with high reproducibility and under high pressure (up to 4000 psi).

Columns: Columns are usually made of polished stainless steel, are between 50 and 300 mm long and have an internal diameter of between 2 and 5 mm. They are commonly filled with a stationary phase with a particle size of 3–10 µm. Columns with internal diameters of less than 2 mm are often referred to as microbore columns. Ideally the temperature of the mobile phase and the column should be kept constant during an analysis.

Detector: The HPLC detector, located at the end of the column detect the analytes as they elute from the chromatographic column. Commonly used detectors are UV-spectroscopy, fluorescence, mass-spectrometric and electrochemical detectors.

Data Collection Devices: Signals from the detector may be collected on chart recorders or electronic integrators that vary in complexity and in their ability to process, store and reprocess chromatographic data

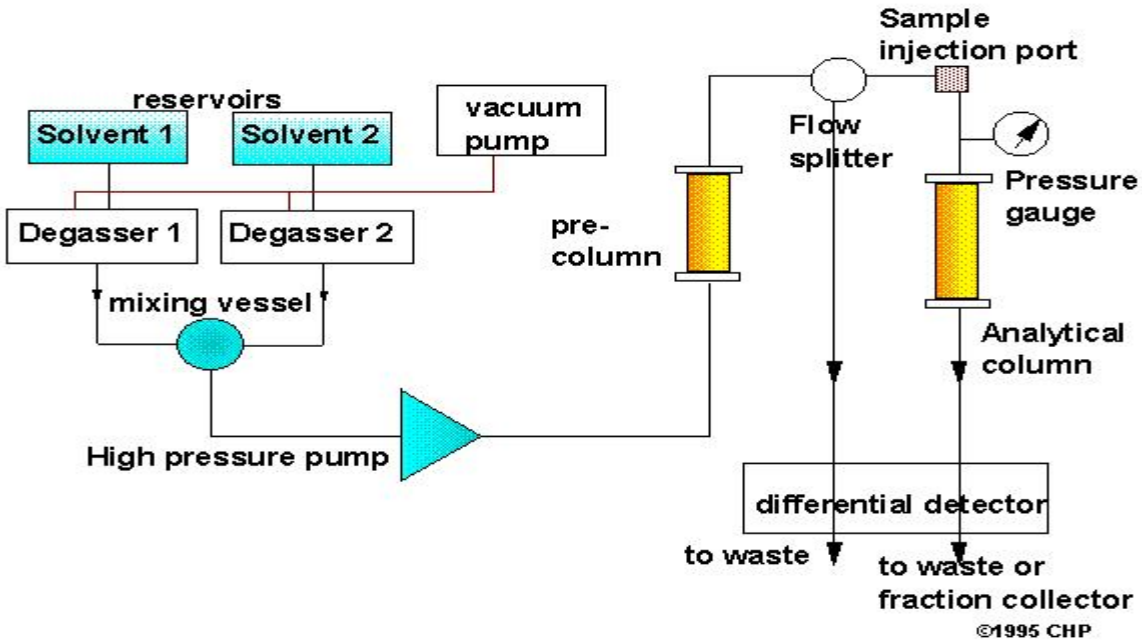


Figure no 11 Schematic Diagram of High Performance Liquid Chromatography

Instrument

Solvent Resorvoir: Mobile phase contents are contained in a glass resorvoir. The mobile phase, or solvent, in HPLC is usually a mixture of polar and non-polar liquid components

The computer integrates the response of the detector to each component and places it into a chromatograph that is easy to read and interpret.

Gas Chromatography

Principle

Gas chromatography runs on the principle of partition chromatography In gas chromatography, the mobile phase (or "moving phase") is a carrier gas, usually an inert gas such as helium or an unreactive gas such as nitrogen. Helium remains the most commonly used carrier gas in about 90% of instruments although hydrogen is preferred for improved separations. The stationary phase is a microscopic layer of liquid or polymer on an inert solid support, inside a piece of glass or metal tubing called a column (an homage to the fractionating column used in distillation). The instrument used to perform gas chromatography is called a gas chromatograph (or "aerograph", "gas separator").The gaseous compounds being analyzed interact with the walls of the column, which is coated with a stationary phase. This causes each compound to elute at a different time, known as the retention time of the compound. The comparison of retention times is what gives GC its analytical usefulness.

Gas chromatography is in principle similar to column chromatography (as well as other forms of chromatography, such as HPLC, TLC), but has several notable differences. First, the process of separating the compounds in a mixture is carried out between a liquid stationary phase and a gas mobile phase, whereas in column chromatography the stationary phase is a solid and the mobile phase is a liquid. Second, the column through which the gas phase passes is located in an oven where the temperature of the gas can be controlled, whereas column chromatography (typically) has no such temperature control.

Instrumentation

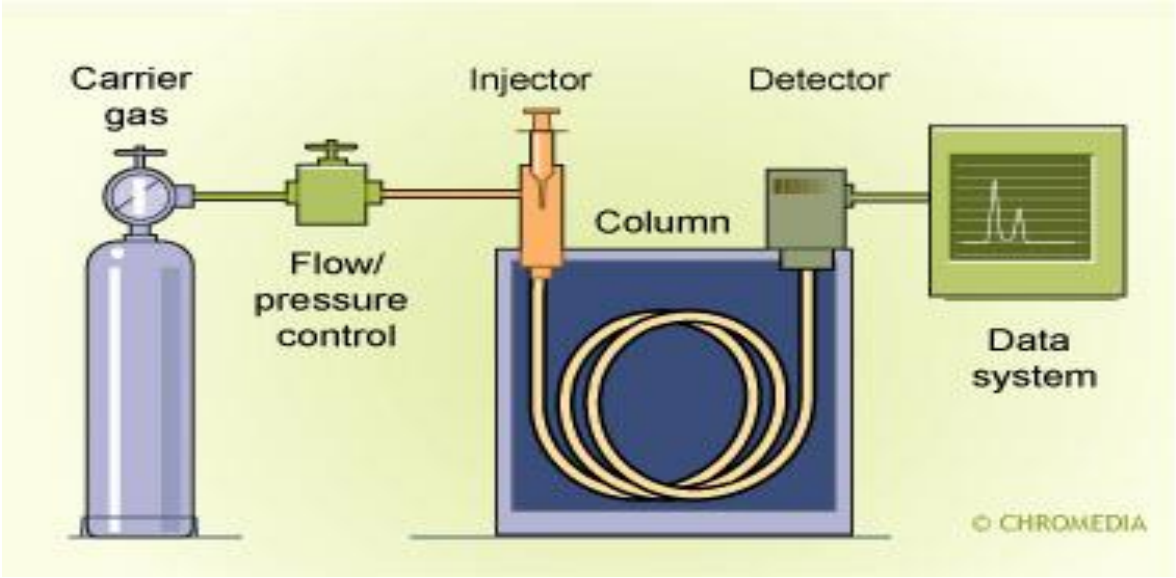


Figure no 12 Schematic Diagram of Gas Chromatography

Many commercial variations are available, basically all gas chromatographs, whether GLC or GSC, consists of six basic components.

1. A carrier gas
2. A sample injection system.
3. The separation column.
4. Detectors.
5. Thermostat chambers for the temperature regulation of column and detectors.
6. An amplification and recorder system.

Carrier Gas: The most widely used carrier gas are hydrogen, helium, nitrogen and air. Hydrogen- More advantage as compared to other gases but is dangerous to use. It has better thermal conductivity, lower density and greater flow rates. Helium - It is used because of its excellent thermal conductivity, inertness, low density and it allows greater flow rates. Nitrogen - It is inexpensive but gives reduced sensitivity. Air - It is used only when the oxygen in the air is useful to the detector or separation. Oxygen is usually avoided since it oxidizes the stationary phase.

IDEAL Conditions For Carrier Gas is as follows: It should be inert and not react with the sample, stationary phase or hardware. It should be suitable for the detector employed and type of sample to be analyzed. It should be readily available in high purity. It should give best column performance consistence with required speed of the analysis. It should be cheap and not cause the risk of fire or explosion hazards.

Sample Introduction System: The sample introduction system is very important. very small amount of sample is used. The sample – reproducible and must vaporize it instantaneously so that the sample will enter the column as a single slug. SOLIDS - samples must be dissolved in volatile liquids for introduction or may be introduced directly if they can be liquefied. LIQUIDS-By using hypodermic syringe through a self-sealing rubber septum into a small inlet chamber, which may be heated to cause flash evaporation.

GASES-Gas samples require a special gas sampling valves for introduction into the carrier gas stream.

Column: In GC, retention of analyte molecules occurs due to stronger interactions with the stationary phase than the mobile phase. This is unique in GC and, therefore, interactions between the stationary phase and analyte are of great importance. The interaction types can be divided into three

broad categories: Dispersive• Dipole• Hydrogen bonding• The sample is separated into its constituent components in the column. Columns vary in length and internal diameter depending on the application type and can be either packed or capillary. Packed columns (typical dimension 1.5 m x 4 mm) are packed with a solid support coated with immobilized liquid stationary phase material (GLC). Capillary columns (typical dimension 30 m x 0.32 mm x 0.1 mm film thickness) are long hollow silica tubes with the inside wall of the column coated with immobilized liquid stationary phase material of various film thickness.

Detectors

The detector responds to a physicochemical property of the analyte, amplifies this response and generates an electronic signal for the data system to produce a chromatogram. Many different detector types exist and the choice is based mainly on application, analyte chemistry and required sensitivity – also on whether quantitative or qualitative data is required. Detector choices include:

1. Flame Ionization (FID)
2. Electron Capture (ECD)
3. Flame Photometric (FPD)
4. Nitrogen Phosphorous (NPD)
5. Thermal Conductivity (TCD) and Mass Spectrometer (MS)

Ideal Characterictics of Detectors The sensitivity should be high and without instability at high sensitivities. The volume should be low so that the compound eluted from the column in a small plug of carrier gas is not diluted further within the detector itself. The response should be rapid and linear with concentration of compound. The response should be unaffected by flow rate of carrier gas and temperature. The response should be fast.

Flame Ionisation Detector Principle*:* Based on the electrical conductivity of gases. At normal temperature and pressure gases acts as insulators but will become conductive of ions if electrons are present. Very small number of ions can be detected on the basis of conductivity.

Working: Hydrogen is added with a capillary jet if it was not used as carrier gas. The mixture is burnt in air (or oxygen) in the detector. The platinum wire serves as one electrode of the cell and the collector is the other. A sufficient potential to collect all ions is used. This detector is remarkably insensitive to the presence of water vapor or air in the carrier gas, and the background current is low so that small quantities can be measured with proper amplification.

Electron Capture Detector

Electron Capture Detector The electron affinity of different substances can be used as the basis for ionization detection known as the electron capture detector. It depends to only those compounds whose molecules have an affinity for electrons. E.g., Chlorinated compounds, alkyl lead, etc. It can also be employed for pesticide analysis (subpicogram) and those which Accept electrons of carrier gas.On the contrary it responds very little to compounds such as hydrocarbons.

Electron Capture Detector ECD detects ions at the exit of the gas from chromatographic column by the anode electrode.(3 H or 63 Ni which emits β particles). Ionization: N 2 (Nitrogen carrier gas) + β (e) = N 2 + + 2e , These N 2 + establish a “base line” X (F, Cl and Br) containing sample + β (e) ◊ X - Ion recombination : X - + N 2 + = X + N 2 The “base line” will decrease and this decrease constitutes the signal. Insecticides, pesticides, vinyl chloride, and fluorocarbons

Electron Capture DetectorOther GC detectors ℵNitrogen-Phosphorous Detector (NPD) Alsoknow as the thermionic detector (TID) or alkali flame detector. It is an FID tweaked for N-P cpds, and organics. ℵ Flame Photometric Detector (FPD) FID tweaked for S containing cpds. ℵPhotoionization Detector (PID) UV ionization of organic analyte, coupled with high voltage cathode and analode results in current proportional to ionized organics.

Thermal Conductivity Detectorworking of Detector: Conductivity of the carrier gas in the presence of an organic compound. The tungsten wires are heated electrically and assume equilibrium conditions of temperature and resistance. Wheatstone bridge arrangement - signal, which is amplified and recorded. The sensitivity is low and affected by fluctuations of temperature and flow rate.Responds to all compounds Adequate sensitivity for many compounds Good linear range of signal Simple construction Signal quite stable provided carrier gas flow rate, block temperature, and filament power are controlled Nondestructive detection Thermal Conductivity Detector

Thermal Conductivity Detector (Kathertometer)

Principle: The thermal conductivity of the gas due to resistance developed by temperature. Electrical power is converted to heat in a resistant filament by the increasing temperature which causes heating. The filament may loose heat by radiation to a cooler surface and by conduction to the molecules coming into contact with it.

Thermostat Chambers: Temperature in GC is controlled via a heated oven. The oven heats rapidly to give excellent thermal control. The oven is cooled using a fan and vent arrangement usually at the rear of the oven. A hanger or cage is usually included to support the GC column and to prevent it touching the oven walls as this can damage the column. The injector and detector connections are also contained in the GC oven. For Isothermal operation, the GC is held at a steady temperature during the analysis. In temperature programmed GC (pTGC) the oven temperature is increased according to the temperature program during the analysis.

Recorder System: The data system receives the analogue signal from the detector and digitizes it to form the record of the chromatographic separation known as the ‘Chromatogram’ The data system can also be used to perform various quantitative and qualitative operations on the chromatogram – assisting with sample identification and quantitation.

CONCLUSION

In this review attempt is made to incorporate various analytical instruments and their principle with their detail instrument analytical Instruments like i.e. Ultraviolet spectroscopy, I.R, Raman Spectroscopy, Flame Spectroscopy, Atomic Absorption Spectroscopy, Nuclear Magnetic Resolution Spectroscopy, Coulometry , High performance liquid chromatography, gas

chromatography. They are used often because of its speed, simplicity and availability. It has applications in various areas chemical and drug analysis.

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