18EI703

Hall Ticket Number:

IV/IV B.Tech (Regular/Supplementary) DEGREE EXAMINATION

November,2022 **Electronics &Instrumentation Engineering**

Seventh Semester **Analytical instrumentation**

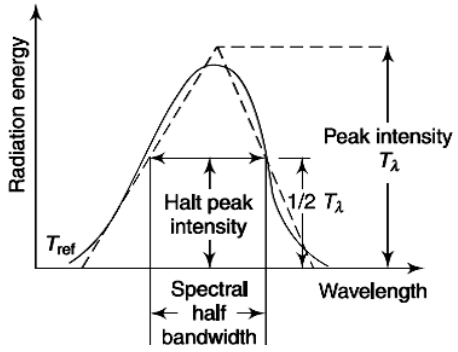
Time: Three Hours **Maximum: 5**0 Marks

Answer Question No. 1 Compulsorily. (10X1 = 10 Marks)

*Answer **ANY ONE** question from each Unit.* (4X10=40 Marks)

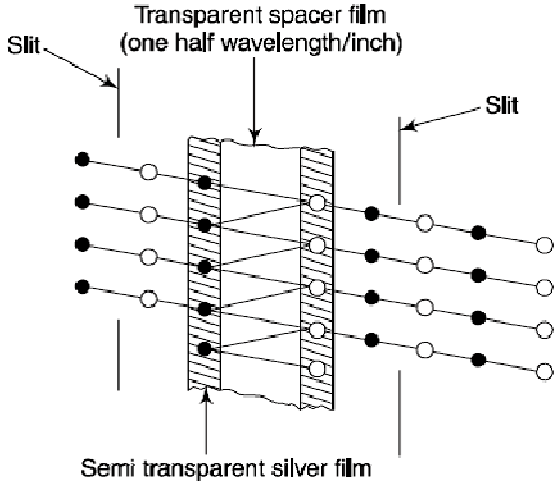
1.	a)	State the Lambert's Law.	CO1(BL1)	
	b)	Define Electromagnetic radiation.	CO1(BL1)	
	c)	List out the examples of UV Sources.	CO1(BL1)	
	d)	What is the purpose of 'Atomizer 'in Flame photometry?	CO2(BL1)	
	e)	Define the term NMR.	CO3(BL1)	
	f)	Give the principle of Fourier Transform IR Spectrometer.	CO3(BL1)	
	g)	What is meant by ionization?	CO4(BL1)	
	h)	Compare the functions of Prisms and Gratings.	CO1(BL1)	
	i)	Give examples of scintillating crystals.	CO4(BL1)	
	j)	List some applications of Gas chromatography.	CO4(BL1)	
Unit – I				
2.	a)	Discuss the optical system of UV and Visible spectroscopic instruments.	CO1(BL2)	**5M**
	b)	Discuss the working of double beam ratio recording spectrometer.	CO1(BL2)	**5M**
		(OR)		
3.	a)	Determine the relation between absorption and concentration.	CO1(BL3)	**5M**
	b)	Explain various detectors in UV and Visible Spectroscopy.	CO1(BL2)	**5M**
Unit – II				
4.	a)	Outline the various detectors used in IR spectroscopy.	CO2(BL4)	**5M**
	b)	Explain the construction and working of clinical flame photometer.	CO2(BL2)	**5M**
		(OR)		
5.	a)	Explain the working of FT-IR spectrophotometer with neat diagram.	CO2(BL2)	**5M**
	b)	Write about Emission and recording systems of flame photometers.	CO2(BL1)	**5M**
Unit – III				
6.	a)	Discuss the constructional details of ESR spectrometer with neat sketches.	CO3(BL2)	**5M**
	b)	Explain the construction and working principle of FTNMR spectrometer.	CO3(BL2)	**5M**
		(OR)		
7.	a)	Illustrate the working of time of flight mass analyzer.	CO3(BL3)	**5M**
	b)	Define Electron spin and how it is used in spectroscopy?	CO3(BL1)	
Unit – IV				
8.	a)	Explain in detail about instrumentation associated with X-ray spectroscopy.	CO4(BL2)	**5M**
	b)	Explain graphically the comparative operation of ionizing chamber, GM counter and scintillation counter.	CO4(BL2)	**5M**
		(OR)		
9.	a)	Outline the working of High Pressure liquid chromatography with neat diagrams.	CO4(BL4)	**5M**
	b)	Write short notes on the following.	CO4(BL1)	**5M**
		(i) Proportional Counter		
		(ii) Solid State Detector.		

1. a) State the Lambert's Law.
   Absorption of light by a sample is proportional to thickness.
   b) Define Electromagnetic radiation.
   Electromagnetic radiation consists of Electric and Magnetic fields perpendicular to each other. The electromagnetic spectrum consists of different regions like Ultraviolet, Visible, Infrared , X-Rays, Radio Waves etc.
   c) List out the examples of UV Sources.
   Tungsten Lamp, Hydrogen-Deuterium discharge lamp, LASER
   d) What is the purpose of 'Atomizer 'in Flame photometry?
   It is used convert sample to gaseous state known as 'aerosol'.
   e) Define the term NMR.
   Nuclear Magnetic Resonance: Nuclei of atoms absorb RF signal under the influence of external magnetic field.
   f) Give the principle of Fourier Transform IR Spectrometer.
   It consists of Michelson Interferometer which generates "Interferogram". By applying Fourier Transform we get Absorption Spectrum.
   g) What is meant by ionization?
   When atoms absorb energy, the electron is removed from it generating Positive Ions, which is known as Ionization.
   h) Compare the functions of Prisms and Gratings.
   Prisms: They work based on the principle of "change in the refractive index of material with wavelength".
   Gratings: The light reflected from highly polished surface with vertical grooves will have interference. Te wavelength having constructive interference is selected.
   i) Give examples of scintillating crystals.
   NAI
   j) List some applications of Gas chromatography.
   Food analysis, Quality control, Forensics, Pollution monitoring etc.
2. a) Discuss the optical system of UV and Visible spectroscopic instruments.
   Ans: It consists of Optical Filters, Monochromators
   Filters:
   (i) Absorption Filters:
   The absorption type optical filter usually consists of color media: color glasses, colored films (gelatin, etc.), and solutions of the colored substances. This type of filter has wide spectral bandwidth, which may be 40–50 m in width at one-half the maximum transmittance. Their efficiency of transmission is very poor and is of the order of 5–25%. It is possible to obtain more selective light filters from colored media by increasing their thickness two, three or more times. Here the transmission of the filter for light of the wavelength isolated is decreased, but there is a simultaneous increase in the selectivity. By using this technique, it is theoretically possible to achieve a very good selectivity, but the fall in transmission efficiency would have to be compensated by suitable amplification of the photocurrent. As absorption type filters do not provide a high degree of monochromaticity required for isolating complex systems, their use is restricted to only very simple type of photometers.



   (ii) Interference Filters:
   Interference filters usually consist of two semitransparent layers of silver, deposited on glass by

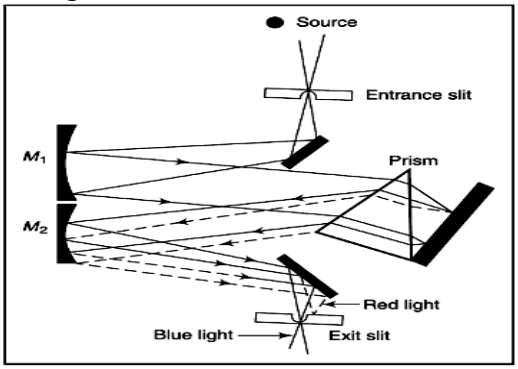
evaporation in vacuum and separated by a layer of dielectric (ZnS or MgF2). In this arrangement, the semi-transparent layers are held very close. The spacer layer is made of a substance which is of low refractive index. The thickness of the dielectric layer determines the wavelength transmitted. Figure 2.17 shows the path of light rays through an interference filter. Some part of light that is transmitted by the first film is reflected by the second film and again reflected on the inner face of the first film, as the thickness of the intermediate layer is one-half a wavelength of a desired peak wavelength. Only light which is reflected twice will be in phase and come out of the filter, other wavelengths with phase differences would cause destructive interference. Constructive interference between different pairs in superposed light rays occurs only when the path difference is exactly one wavelength or some multiple thereof. The relationship expressing a maximum for the transmission of a spectral band is given by $n\lambda = 2d(n)\sin\theta$.



Monochromators:

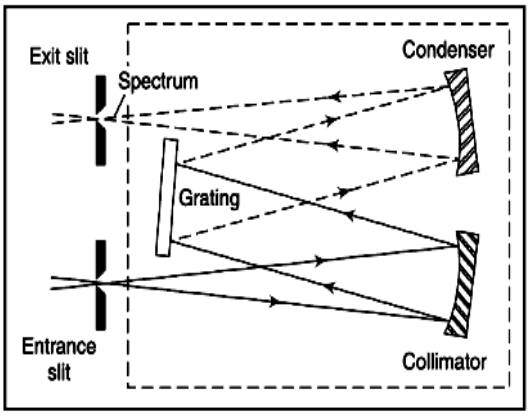
(i) Prisms:

Principle: Light of different wavelengths is bent by different angles when it travels from one medium to another medium (of different refractive indices). So light of different wavelengths are separated and can be selected. When a mirror is placed on the other side of prism light is reflected back and passes through the prism second time. This is called Littrow mounting. This will cause better separation. Prism made up of glass is used visible region and the one with Quartz is used in UV region.



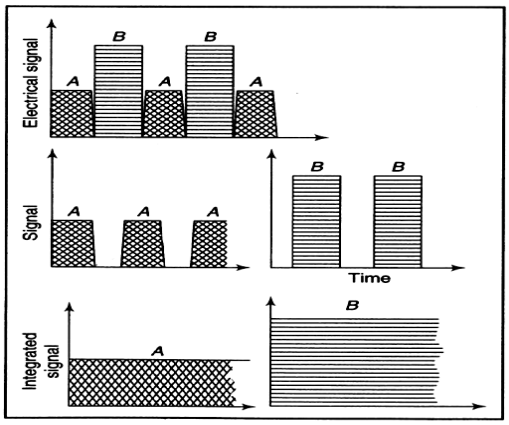
(ii) Diffraction Gratings:

It consists of a highly polished surface upon which number of grooves are present. The light rays reflected from these grooves have either constructive or destructive interference depending on the path difference. Constructive interference occurs when the path difference is an integral multiple of wavelength. $d(\sin i+\sin r)=n\lambda$. Here i is the incidence angle and r is the reflection angle.



b) Discuss the working of double beam ratio recording spectrometer.

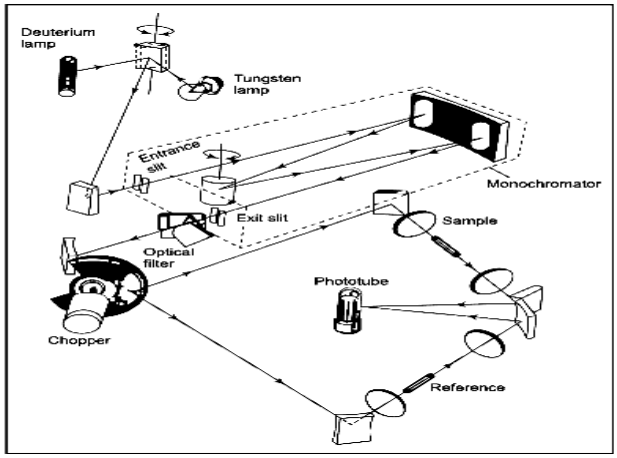
Double-beam Ratio-Recording Spectrometer:



Operation:

In this spectrometer light from source is split into two beams and they are passed through sample and reference solutions alternately by using a chopper. As shown in Figure 1 the output from the detector is an alternating electrical signal. The signal denoted as '**A**' is the due the sample beam and is of smaller amplitude due to absorption of light by the sample solution and the signal denoted by 'B' is due to the reference beam and is larger since the reference solution doesn't absorb light. The signals '**A'** and '**B'** are separated and integrated using an electronic integrator. The ratio of these integrated signals is calculated to determine either Transmittance or Absorbance.

Figure shows the optical diagram of the spectrometer. It consists of interchangeable Tungsten and Deuterium Discharge lamp. A sector mirror is used to pass the light through reference and sample beams alternately. Monochromator and optical filter are used for wavelength selection.



(OR)

3. a) Determine the relation between absorption and concentration.

b) Explain various detectors in UV and Visible Spectroscopy.

(i) Photovoltaic of Barrier layer cell:

The photovoltaic cell consists of a semiconductor like Selenium which absorbs light energy and generates electron-hole pairs. The electrons and holes constitute current and is proportional to the light intensity. The cell consists of selenium deposited on Iron base and a glass window to allow light and the entire set up is enclosed in a plastic case.

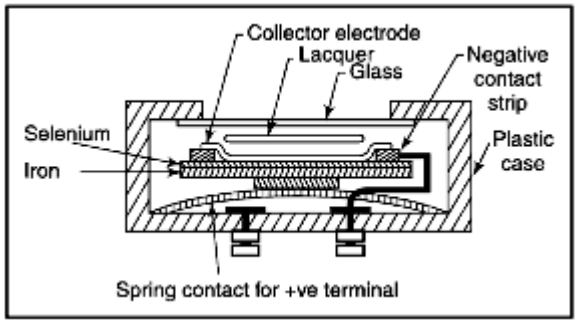
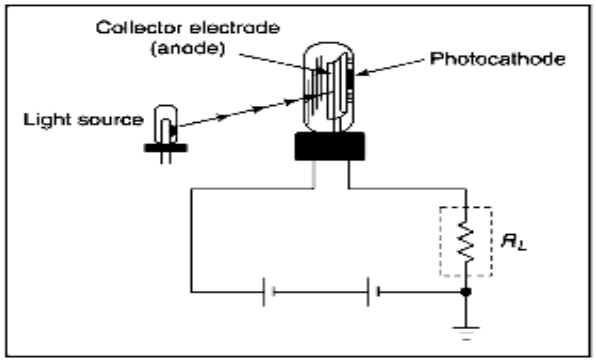


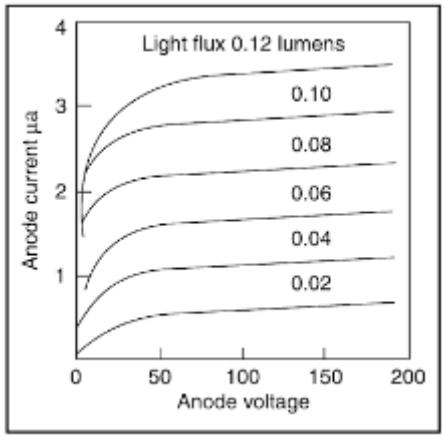
Figure 1

(ii) Photo-emissive cells:

It consists of a semi cylindrical cathode and a wire anode. The cathode is coated with a photo-emissive material. When light falls upon the cathode the photo-emissive material absorbs light energy and emits electrons. These electrons are collected by the anode resulting in a current which is proportional to the light intensity.

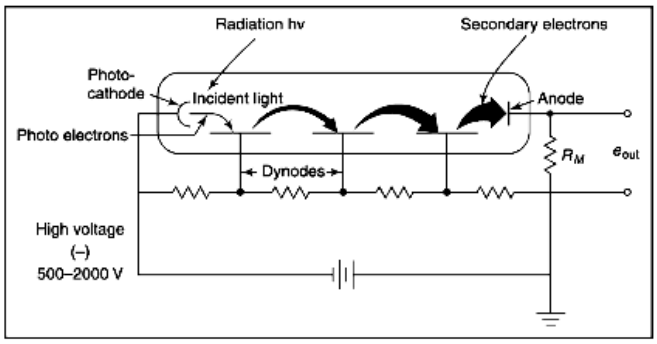


The current increases with applied voltage and light intensity (lumen) as shown below.



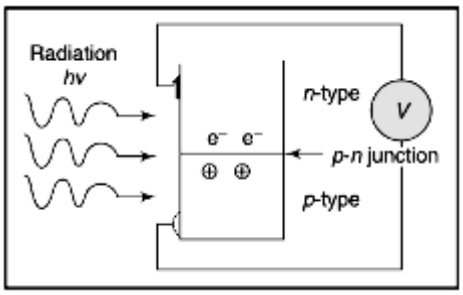
(iii) Photomultiplier tube (PMT):

In a photomultiplier tube along with cathode there are 9 dynodes. The first dynode is +90V more positive than cathode and second dynode is +90V more positive than first one and so on. When light is incident on cathode it generates electrons. These electrons travel towards first dynode and strike it to generate few more electrons. Like this carrier multiplication takes place from dynode to dynode. After 9^{th} dynode huge number electrons are generated and current is large enough to be indicated by a galvanometer and there is no need of amplifier.



(iv) Semiconductor Detector:

When light is incident near the junction of a p-n junction diode electron hole pairs are generated. These electron-hole pairs constitute some current.



4. a) Outline the various detectors used in IR spectroscopy.

Quantum detectors:

(i) Photoconductive cells:

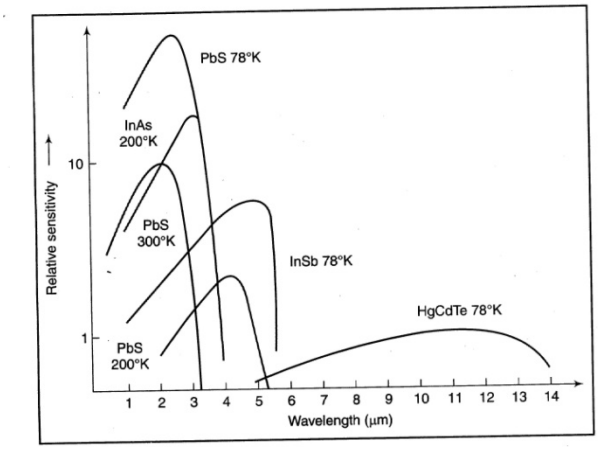
Elecrical resistors whose resistance decreases with intensity of light. Constructed from thin layer of semiconductor like Lead Sulphide (PbS) of Lead Telluride (PbTe) supported on a backing medium like glass and sealed into an evacuated glass envelope.

(ii) Photodetectors:

Semiconductors exhibit photoelectric effect.

Ex: PbS,InSb,CMT etc

Detectors must be cooled to prevent back ground thermal excitation.



(iii) Photodiode:

InGaAs photodiode arrays are used in near IR spectroscopy. Vaccum sealed and antireflective coated windows are present. Cooling arrangement reduces dark current and facilitates longer exposure times.

Thermal detectors:

(i) Thermocouple:

Consists of two junctions formed by two dissimilar metals. These are called as cold and hot junctions. When there is a temperature difference between these junctions emf is generated. This is called as Seebeck effect.

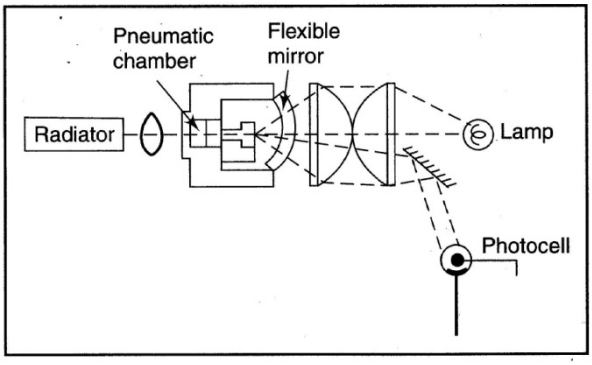
(ii) Bolometer:

Consists of a Pt- strip in an evacuated glass vessel with transparent window in the IR range. Irradiation causes an increase in the resistance of the metal strip, which when placed in a

Wheatstone bridge produces output voltage.

(iii) Pneumatic detector:

Contains a chamber of Xenon, a gas of low thermal conductivity. This chamber is sealed at its front by a blackened receiver. The rear wall is a flexible membrane with a mirrored surface on its rear side. A rise in the temp of the chamber produces a corresponding rise in pressure and therefore distortion in mirror diaphragm. Light from a lamp inside detector housing can be focused on the diaphragm, which reflects light onto a photocell.



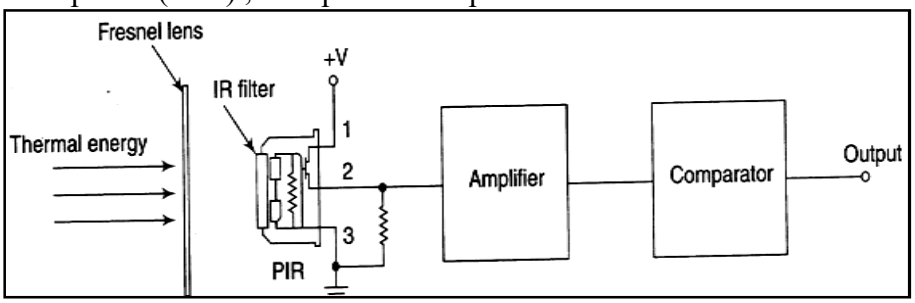
(iv) Pyroelectic detector:

Ferro electric crystals get polarized in a well defined direction known as polar axis. Degree of polarization is temperature dependent. Charge gets accumulated on the faces normal to the polar axis which is proportional to polarization. This is called "pyro-electric effect".

Signal processing for Pyro-electric detector:

Has two sensing elements and cancels the signals caused by vibration.

A amplifier (FET) , Comparator are present.

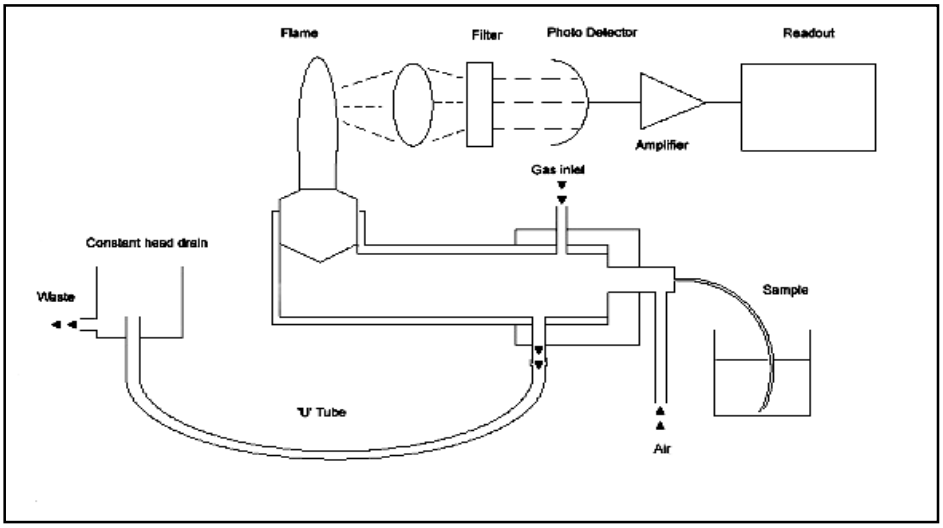


b) Explain the construction and working of clinical flame photometer.

The basis of low temperature flame photometry is the same as that of the simple quantitative analytical flame test. This exploits the fact that compounds of the alkali and alkaline earth metals are thermally dissociated into atoms at the temperature of a Bunsen burner flame and that some of these atoms produced are further excited to a higher energy level. When these 'excited' atoms return to the ground state, they emit radiation, which for the elements of these two groups lies mainly in the visible region of the electromagnetic spectrum. The estimation of the alkali metals by flame photometry is by far its most important application in routine chemical analysis. For this widespread requirement, low temperature flame photometry provides the most reliable and convenient procedure available.

This technique has considerable appeal in the clinical chemistry field as it provides a rapid and reliable means of estimating sodium, potassium and lithium in body fluids. The methodologies of such analyses are well known and indeed well established and shall therefore not be discussed in this page.

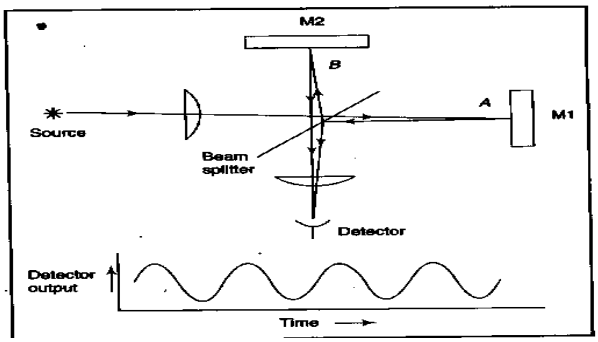
However the estimation of the alkali and alkaline earth metals is commonly required in a sample matrix which does not lend itself to simple and direct analysis involving only a dilution step, e.g. sodium in fuel oil.



(OR)

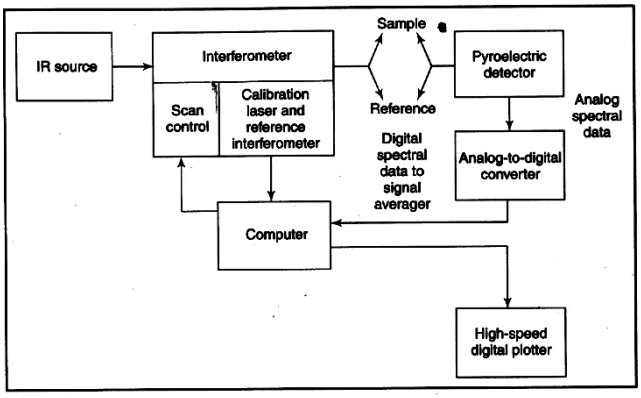
5. a) Explain the working of FT-IR spectrophotometer with neat diagram.

Heart is two beam interferometer (Michelson interferometer). Radiation is split into two beams and path difference is introduced. When the path difference is an integral multiple of λ constructive interference occurs and intensity is maximum. When mirror M is applied with displacement then path difference changes and intensity changes. The overall detector output as function of time will be the sum of the wave for each frequency component. This represents Fourier Transform and spectral information can be extracted by applying Inverse Fourier Transform.



(3M)

Block Diagram:



b) Write about Emission and recording systems of flame photometers.

A flame photometer has three essential parts.

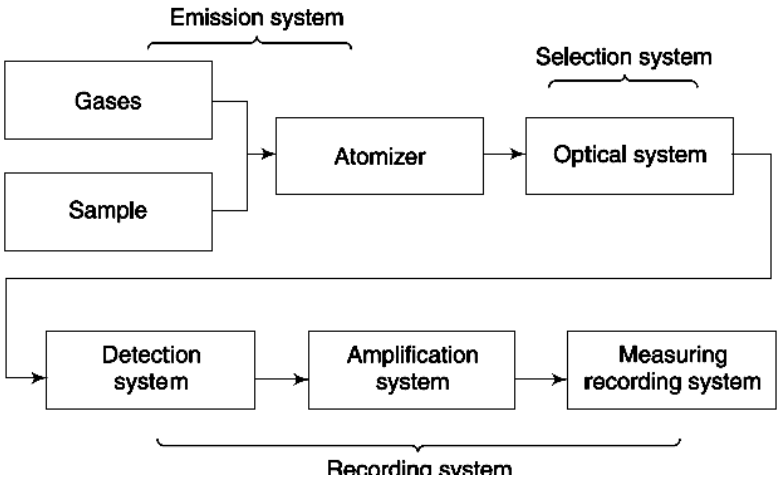
Emission System: It consists of the following:

(i) Fuel gases and their regulation: comprising the fuel reservoir, compressors, pressure regulators and pressure gauges.

(ii) Atomiser: consisting, in turn, of the sprayer and the atomisation chamber, where the aerosol is produced and fed into the flame.

(iii) Burner: receives the mixture of the combustion gases.

(iv) Flame: the true source of emission.



Unit – III

6. a) Discuss the constructional details of ESR spectrometer with neat sketches.

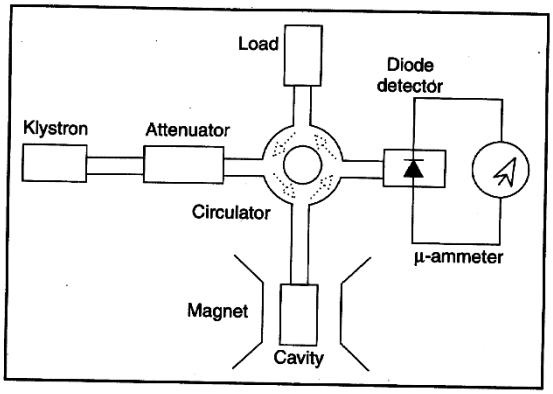
ESR Spectrometer:

Klystron tube: Generator Microwaves

Circulator: Circulates Microwaves from source to cavity then to detector then finally to load.

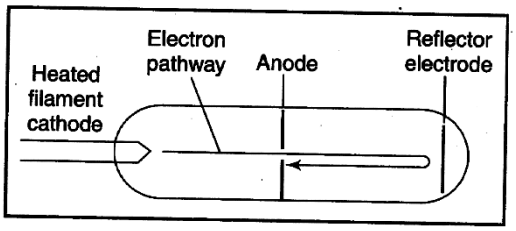
Diode detector: Produces current proportional to Microwaves.

Cavity: Sample is mounted in the cavity



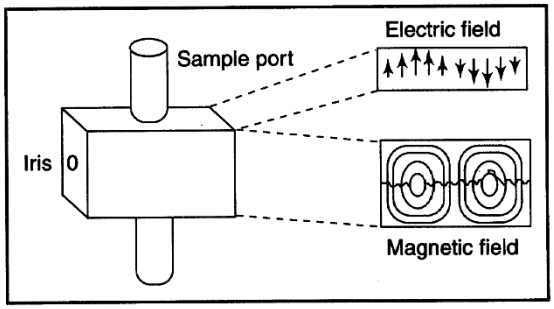
Klystron tube:

Electrons are emitted by cathode and they travel towards anode with velocity and pass trhough the hole and get reflected back by reflecting electrode. This genets Microwaves. Distance is used for coarse frequency adjustment and Reflector voltage for Fine control.

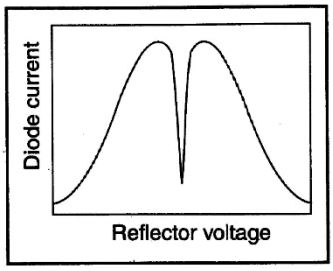


Cavity:

It is a rectangular metal box exactly one wavelength in length. Resonance occurs when the wavelength of MWs equals cavity length.



Diode current vs Reflector voltage:



Dip at cavity resonant frequency.

b) Explain the construction and working principle of FTNMR spectrometer.

Pulsed Fourier Transform Spectrometer:

A pulse of Magnetic field or RF signal is applied through a coil. The nuclei are excited and will precess out. When the pulse is over the nuclei will precess in and the nuclear magnetic moment cuts the coil generating a Free Induction Decay (FID) signal. The frequency of the FID signal is equal to the precession frequency of the nucleus. Since the sample consists of different elements the resulting signal is a sumof different frequency FID signals. The frequencies and their amplitudes give the type of element and its concentration. The frequency components can be obtained by applying Fourier Tranformation.

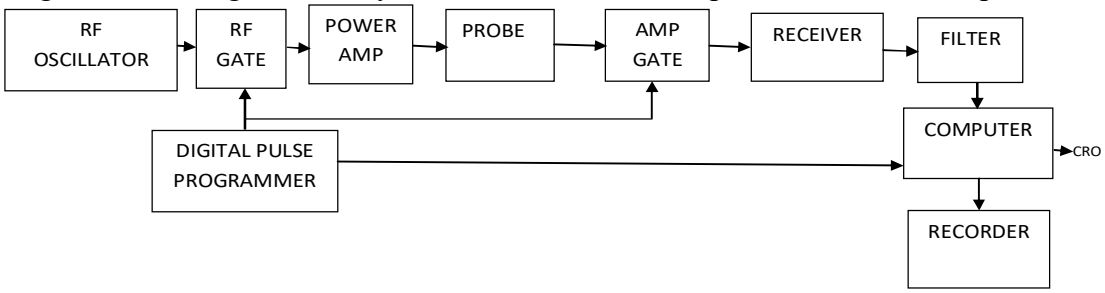
The block diagram of the FTNMR is as shown below.

RF Oscillator: Generates RF signal

RF Gate: Allows RF signal for a specific duration

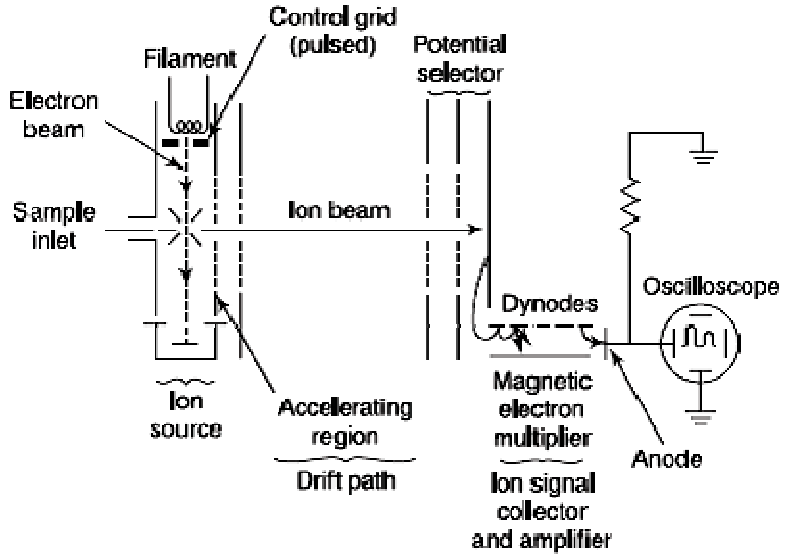
Probe: Houses coils and sample

Digital Pulse Programmer: Synchronizes RF Gate, Amplifier Gate and Computer.



(OR)

7. a) Illustrate the working of time of flight mass analyzer.



parts: • An electron gun for the production of ions. • A grid system for accelerating ions to uniform velocities in a pulsed mode. • An evacuated tube, called the drift tube. • An ion detector and suitable electronic circuitry for translating the time-dependent arrival of ions of different velocities into a time base that is related to mass number.

If L is the length of the drift tube in centimetres and t is transit time in micro-seconds, for singly charged ions of mass m and constant energy Ve, then

$$t = L\,[m/2\,Ve]^{1/2} \qquad (9.7)$$
$$= L\,[m/e \times 1/2V]^{1/2}$$
$$m/e = 2V/L^2 \times t^2$$

If the detector is sensitised for a period Δt at time t, the resolution $\Delta m/m$, for constant energy, is given by

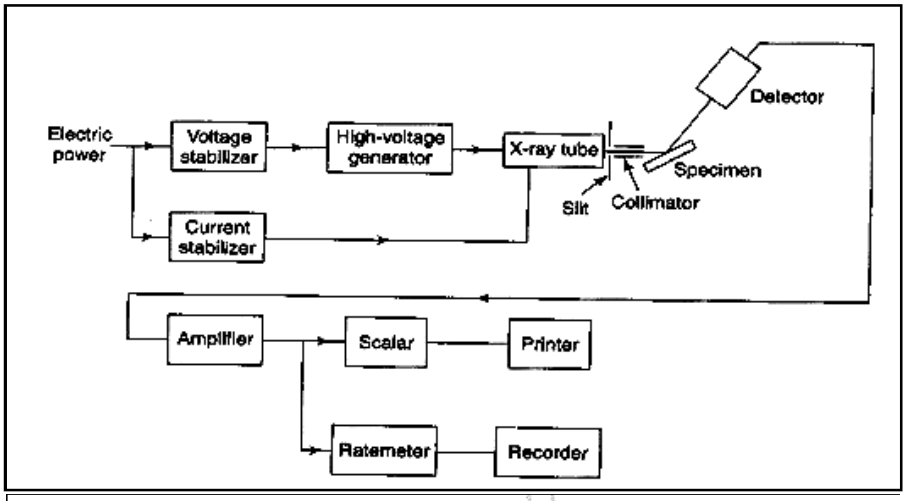
$$\text{Resolution} = \Delta m/m = 2\,\Delta t/t$$

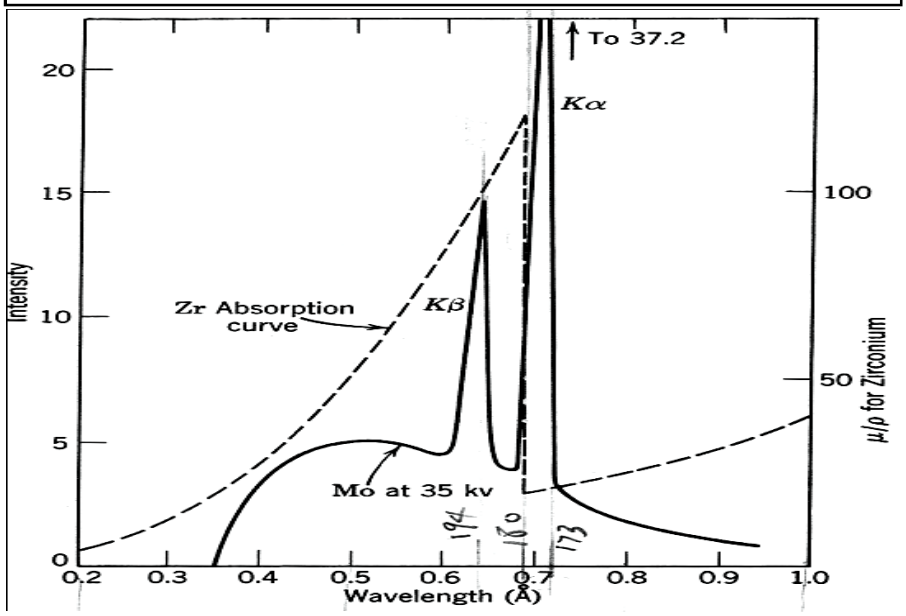
b) Define Electron spin and how it is used in spectroscopy?
CO3(BL1)

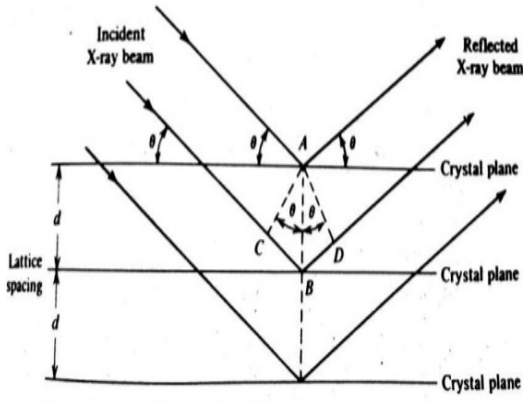
8. a) Explain in detail about instrumentation associated with X-ray spectroscopy.
Components of X-ray Spectrometer:

(i) X-ray source
(ii) Collimator
(iii) Monochromator
(iv) Readout device

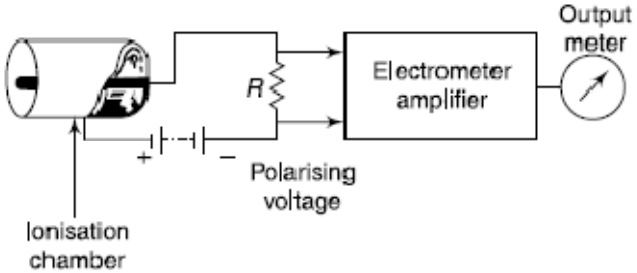
(i) X-Ray Source:
X-Ray source consists of cathode and anode. The electrons from cathode hit the anode coated with heavy metals like Molybdenum. Molybdenum emits X-Rays.
(ii) Collimator:
Used for increasing the resolution of X-Rays.
Monochromator:
It is used for selection of wavelengths.
They are of two types: Filter or Analyzing crystal.
Filter:
Zirconimum can be used as filter for x-rays from a Molybdenum target if Kβ is to be removed.
Analyzing Crystal:
Crystal having planes diffract x-rays of different wavelengths depending on the angle of incidence given by Bragg's Law.
$n\lambda=2d\sin\Theta$







b) Explain graphically the comparative operation of ionizing chamber, GM counter and scintillation counter.

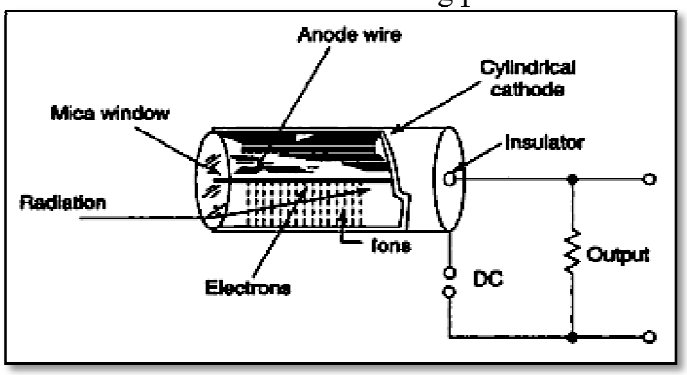


An ionisation chamber consists of a chamber which is gas filled and is provided with two electrodes. A material having a very high insulation resistance, such as polytetrafluoroethylene is used as the insulation between the inner and outer electrodes of

the ion chamber. A potential difference of a few hundred volts is applied between the two electrodes. The radioactive source is placed inside or very near to the chamber. The charged particles moving through the gas undergo inelastic collisions to form ion pairs. The voltage placed across the electrodes is sufficiently high to collect all the ion pairs. The chamber current will then be proportional to the amount of radioactivity in the sample. lionisation chambers are operated either in the counting mode, in which they respond separately to each ionising current, or in an integrating mode involving collection of ionisation current over a relatively long period.

Geiger-Muller Counter: It is operated high voltages. At such high voltages the electrons and ions generated due radiation particles move towards anode at high velocities and collide with gaseous atoms and further generates ions and electrons. The electrons thus generated strike the anode material and generate photons. These photons further cause ionization. Due to this the whole chamber is filled within a microsecond.

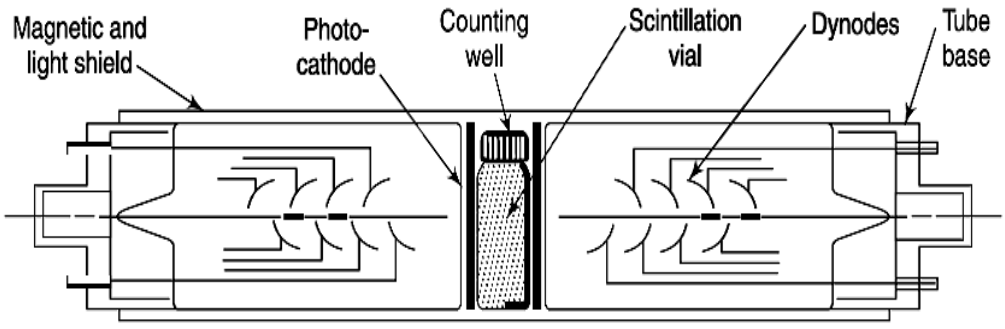
Dead time: Since mobility of ions is smaller they form a sheath around anode making the counter insensitive to fresh ionizing particles.



Scintillation is the process of turning radioactive energy (e.g. the energy associated with a beta particle) into light using a 'scintillator'. A scintillator, thus, is a substance which produces minute flashes of light in the visible or near ultraviolet range, when it absorbs ionising radiation. In such cases, the number of fluorescent photons is proportional to the energy of the radioactive particle. The flashes occur due to the recombination and de-excitation of ions and excited atoms produced along the path of the radiation. The light flashes are of very short duration and are detected by using a photomultiplier tube, which produces a pulse for each particle. A scintillator along with the photomultiplier tube is known as a scintillation counter. The following are the two types of scintillators:

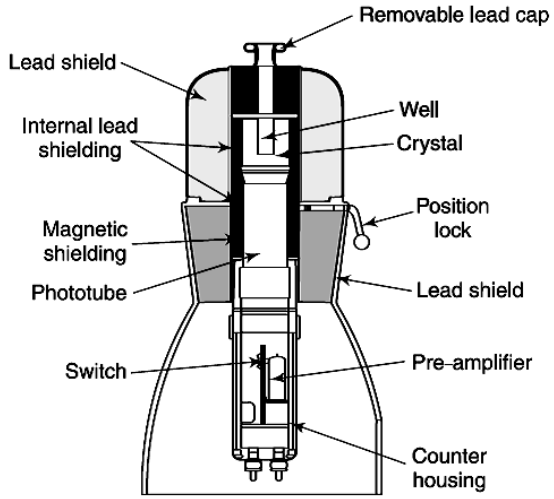
• Liquid Scintillators
• Solids Scintillators

Liquid Scintillators: These are used for lowenergy β− emitters such as 14C, 35S and 3He. The radioactive material is mixed into a scintillation fluid referred to as cocktail. Liquid scintillation fluids are organic molecules dissolved in an organic solvent. The mixture is placed between two photomultipliers which record the light produced. Figure 15.7 shows the simple arrangement of a liquid scintillation counter. A count is recorded only when a pulse of light is simultaneously detected by both PMTs, thus acting as a coincidence counter. This helps to reduce back ground noise.



Solid Scintillators: These are used for high-energy β− emitters such as 203Hg, 59Fe, 65Zn and 109Cd. The most commonly used solid scintillator is NaI (Sodium Iodide). Here, the

radioactive sample is placed in a well (Figure 15.8) cut out of a crystal, whereas the photomultiplier tube is mounted on one face of the crystal. Each β– particle produces several thousand photons of light (wavelength: 400 nm) as it passes through the crystal, which are counted.



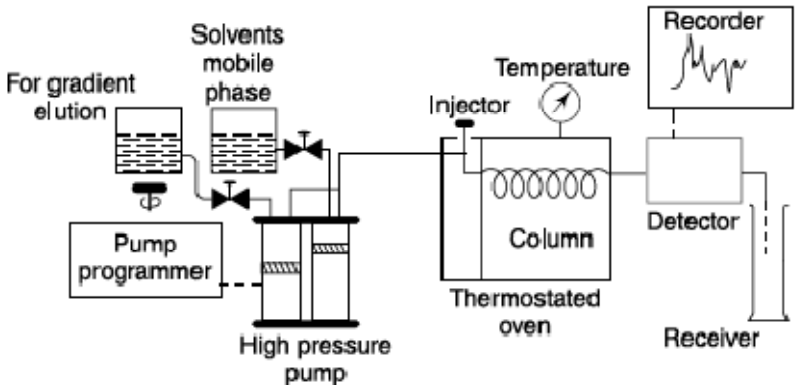
CO4(BL2)

(OR)

9. a) Outline the working of High Pressure liquid chromatography with neat diagrams.

- A high-pressure pump system to force the liquid mobile-phase through the column.
- Gradient elution or solvent programmer.
- The sample injection system.
- The column.
- The detection system including display or recording devices.

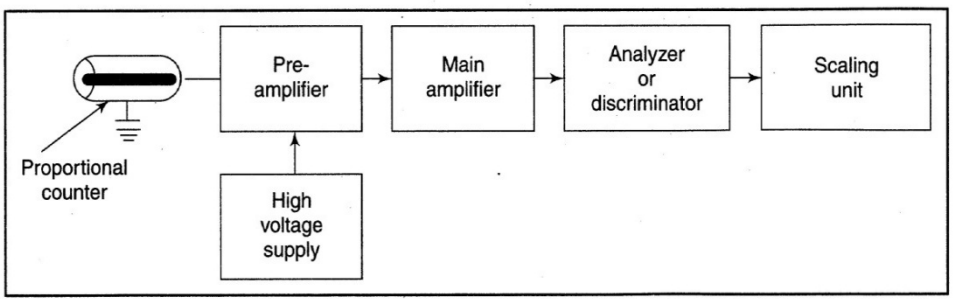
As in other chromatographic techniques, the sample is introduced into the column with the help of a sample injection system. Various components of the sample are fractionated during their passage through the column. The detection system senses these components as they elute from the column and produces a signal proportional to the amount of solutes passing through the detection system. The detector determines what separation has taken place and provides data permitting the qualitative and quantitative evaluation of the results. This can be accomplished by simply recording the response of the detector in the form of a chromatogram and/or with the help of a data handling equipment. The individual sample components separated in the column can also be collected.



b) Write short notes on the following.

(i) Proportional Counter

Proportional counter is operated in between the Ionization chamber and Geiger-Muller counter. In this counter output is proportional to the applied voltage. It has continuous gas supply and hence life is unlimited. Pre-amplifier is of unity gain. Main amplifier has a large gain. Analyzer is a single channel pulse height analyzer.



(ii) Solid State Detector.

Consists of pure Ge placed in between p-type and n-type Ge. When radiation falls upon intrinsic Ge it generates electron hole pairs which generate current.