

**GOVERNMENT OF TAMILNADU
DIRECTORATE OF TECHNICAL EDUCATION
CHENNAI – 600 025**

STATE PROJECT COORDINATION UNIT

Diploma in Instrumentation and Control Engineering

Course Code: 1042

M – Scheme

**e-TEXTBOOK
on
INDUSTRIAL INSTRUMENTATION
for
IV Semester DICE**

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DETAILED SYLLABUS
34244 - INDUSTRIAL INSTRUMENTATION (M-Scheme)

Unit I – COMPARATORS

Pages 3 - 12

Introduction -Types - Mechanical Comparators - Dial Gauge – Reed type comparator - Optical comparators - Optical lever - Cooke Optical Comparator - Zeiss ultra optimeter - Electrical Comparator - Electronic comparator - Pneumatic Comparators - Solex Pneumatic Comparator - Principle of operation, construction, advantages and disadvantages of the above comparators.

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Unit III - MEASUREMENT OF FORCE, TORQUE AND SHAFT POWER

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Torque Measurement: Definition - Principle of operation and construction of - Gravity balance method – Optical torsion meter – Electrical torsion meter – Strain gauge torsion meter.

Shaft Power Measurement: Definition- Principle of operation and construction of - Prony brake Dynamometer – Rope Brake Dynamometer – Fluid Friction (Hydraulic) Dynamometer – Eddy current Dynamometer – D.C Dynamometer.

Unit IV - MEASUREMENT OF pH & GAS ANALYSIS

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pH: Definition - Electrodes - Principle of operation and construction - Hydrogen electrode - Calomel electrode - Quinhydrone electrode - Antimony electrode - Glass electrode.

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Unit V - CHROMATOGRAPHY AND SPECTRAL METHOD OF ANALYSIS

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Detectors: Principle of operation and Construction of TCD, FID, FPD, ECD.

Spectral Analysis: EMR Spectrum - Beer's law - IR/UV spectrophotometry - General description - range of IR/UV radiation - measurement of IR/UV radiation - Instrumentation - IR/UV radiation sources - monochromator - Sample handling

REVISION AND TEST

10 Hrs.

TEXT BOOK:

1. A.K.Sawhney and Puneet Sawhney, "Mechanical Measurements and Instrumentation & Control", Dhanpat Rai & Co (P) Ltd, 12th edition 2001.

REFERENCE BOOKS:

1. R.K.Rajpat, "Mechanical measurements and Instrumentation" S.K.Kataria & Sons, New Delhi- 3
2. Gurdeep R Chatwal and Sham K. Anand, "Instrumentation Methods and Chemical Analysis"- Himalaya Publishing House

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34244 - INDUSTRIAL INSTRUMENTATION (M-Scheme)

UNIT I – COMPARATORS

Introduction – Types - Mechanical Comparator- Optical Comparator - Optical Lever- Cooke Optical Comparator - Zeiss Ultra Optimeter - Electrical Comparator - Electronic Comparator- Pneumatic Comparator - Solex Pneumatic Comparator - Principle of Operation, Construction, Advantages and Disadvantages of the above Comparators.

1. Introduction:

Comparator is an instrument used for comparing the dimensions of a component with a standard.

The purpose of a comparator is to detect and display the small difference between an object and the standard.

S. No.	COMPARATOR	MEASURING INSTRUMENT
1.	It is used to compare dimensions of parts with working standard.	It is to measure the actual dimensions of the parts.
2.	The readings are magnified	Readings are not magnified
3.	Fast and accurate, suitable for mass production	Time consuming, so not suitable for mass production.
4.	Accuracy is independent of operator skill.	Accuracy is dependent of operator skill.

Table 1.1

1.1 USES OF COMPARATOR

- To inspect newly purchased gauges.
- In mass production, where components are to be checked for their dimension.
- As laboratory standard for correction and inspection of working gauges.
- As working gauges.

1.2 TYPES OF COMPARATOR

- Mechanical comparator.
- Optical comparator.
- Electrical and Electronic comparator.
- Fluid displacement comparator.
- Pneumatic comparator, etc.

1.3 MECHANICAL COMPARATORS

Mechanical comparators utilize mechanical methods for magnifying the difference between object size and the standard. Their magnification range lies between 250 to 1000. The following comparators are few examples for mechanical comparator.

- i) Dial indicator (or) Dial gauge.
- ii) Reed type comparator.
- iii) Sigma comparator.
- iv) Johansson 'Mikrokator' etc.

1.3.1 DIAL GAUGE

A dial gauge consists of plunger, rack and pinion arrangement, big dial, small dial, scale locking screw and a pointer.

The dial gauge is initially calibrated with the standard.

Now, when the object to be checked is kept (moved) below the plunger, the plunger moves linearly upward or downward, if there is any difference between the standard and the object.

This movement of the plunger is further magnified by the rack and pinion arrangement and is indicated on the big dial by a movable pointer.

The big dial is graduated (divided) into 100 divisions, so that each division measures 0.01mm. The small dial indicates number of revolutions of the big dial.

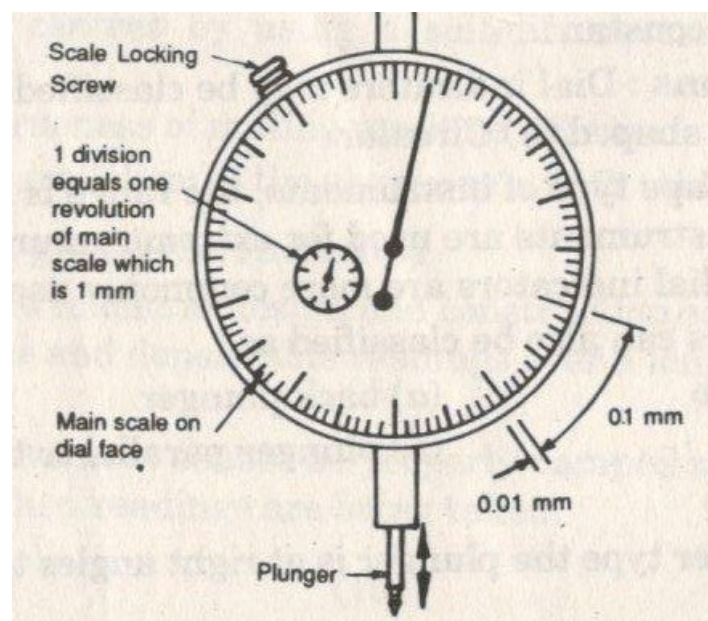


Figure 1.1 Dial Gauge

1.3.2 REED TYPE COMPARATOR

It consists of a fixed block (A) and a moving block (B). The fixed block (A) is rigidly fixed the gauge head case . The moving block (B) carries the gauging spindle. The moving block (B) is horizontally connected with the fixed block (A) by reeds (C). A vertical reed is attached to each block with upper ends joined together by a floating block (D). Above which a pointer is connected. The pointer moves on a calibrated scale.

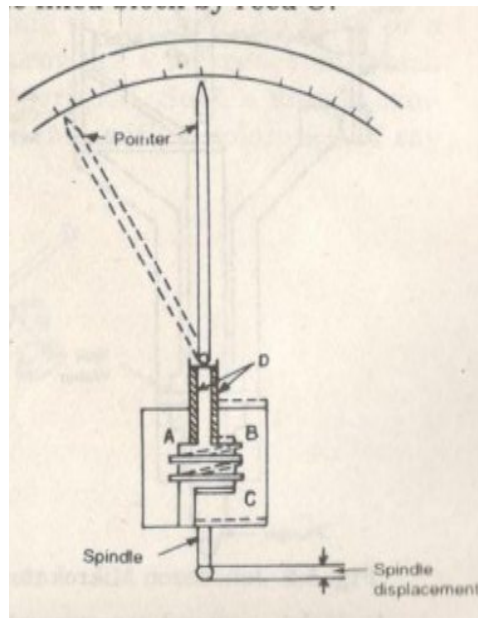


Figure 1.2 Reed Type Comparator

Any linear motion of the spindle moves the moving block vertically causing the vertical reed on the moving block to slide past the vertical reed on the fixed block. This causes the vertical reeds swing through an arc. The amount of swing is proportional to the distance, the moving block has moved. The scale may be calibrated by means of slip gauges to indicate any deviation from an initial setting. It has 0.25×10^{-3} mm / scale division sensitivity.

1.3.4 ADVANTAGES OF MECHANICAL COMPARATORS

- i) Very cheap.
- ii) Have linear scale.
- iii) Robust and easy to handle.
- iv) Compact and portable.
- v) Do not require external supply (electricity, air etc.)

1.3.5 DISADVANTAGES OF MECHANICAL COMPARATORS

- i) Less accuracy (due to moving parts)
- ii) Slackness in moving parts reduces accuracy further.
- iii) Limited range.
- iv) Possibility for parallax error.
- v) Sensitive to vibrations.

1.4 OPTICAL COMPARATORS

Optical comparators are usually based on the principle of reflection of light on a mirror. Here instead of pointer, shadow of the image is projected on a graduated scale to indicate the comparative measurements.

TYPES OF OPTICAL COMPARATOR

- i) Optical Lever Comparator.
- ii) Cooke's Optical Comparator.
- iii) Zeiss Optical Comparator.
- iv) Zeiss Ultra Optimeter, etc.

1.4.1 OPTICAL LEVER COMPARATOR

If a beam of light (OA) is directed on to a mirror, it will be reflected on to the screen at 'B' as a dot. The angle at which the beam strikes the mirror is equal to the angle ' θ ' at which the beam is reflected from the mirror.

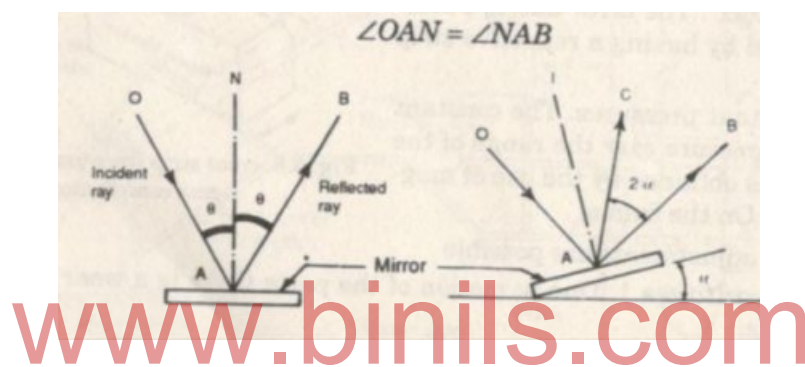


Figure 1.3 Optical Lever Comparator

When the plunger moves upward vertically causing the mirror to tilt by an angle ' α ', then the reflected light beam moves through an angle ' 2α ', which is twice the angle of tilt produced by plunger movement.

The illuminated dot moves to 'B', thus the linear movement small 'h' of the plunger produces a movement of the dot equivalent to the distance CB on the screen.

It is also understood that the magnification can be increased by increasing the distance between screen and the tilting mirror.

1.4.2 COOKE OPTICAL COMPARATOR

The main parts of Cooke Optimeter are

1. Plane mirror 2. Magnifying lever 3. Hinge 4. Plunger 5. Objective lens
6. Measuring table 7. Base and 8. Screen.

Any movement of the plunger tilts the mechanical lever about its hinge. Hence the lever causes small mirror to tilt about the hinge. The hinge of the mirror is in the optical axis of the objective lens. So the reflected ray falls on a circular scale provided at the screen. The scale has a common center with the mirror.

The scale lies in the principal focus of the objective lens. So that light can be sharp along the whole scale.

The total magnification of the comparator is the multiplication of mechanical and optical amplifications.

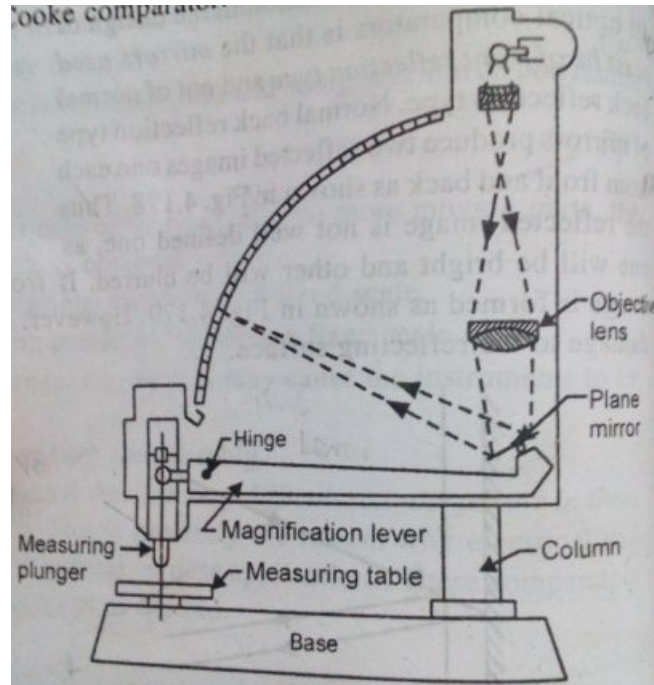


Figure 1.4 Cooke's Optical Comparator

1.4.3 ZEISS ULTRA OPTIMETER

The optical system of Zeiss Ultra Optimeter involves double reflection of light and thus gives higher degree of magnification.

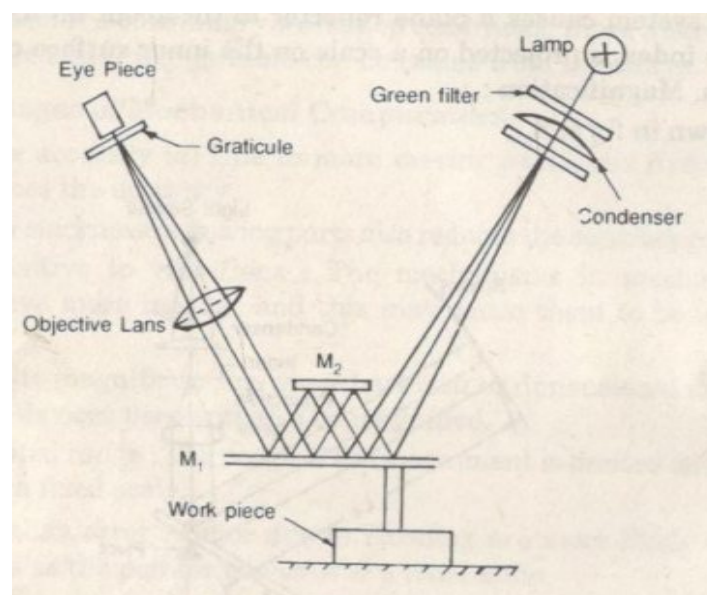


Figure 1.5 ZEISS ULTRA OPTIMETER

CONSTRUCTION

Light rays from a lamp falls on a green filter. This green filter filters all rays except green ray. The green light passes to a condenser, which projects it on to a movable mirror M1. It is the reflected to another fixed mirror M2 and then back again to first movable mirror M1. An objective lens brings the reflected beam from the first mirror (M1) to a focus at a transparent graticule. The graticule consists of a precise scale which is viewed by an eye piece.

OPERATION

Any dimensional variations in the object will tilt the movable mirror M1, resulting in displaced focus point of the reflected beam on the transparent graticule. Thus the readings can be taken by viewing the graticule through the eye piece.

1.4.4 ADVANTAGES OF OPTICAL COMPARATORS

- i) High accuracy (because of fewer moving parts).
- ii) No parallax error.
- iii) High magnification.
- iv) Less weight (optical lever is weightless).
- v) Illuminated scale (helps in taking readings easily).

1.4.5 DISADVANTAGES OF OPTICAL COMPARATOR

- i) Depends on external electrical supply.
- ii) Bulky and expensive.
- iii) Inconvenient for continuous use (because scale to be viewed through eyepiece).
- iv) Heat from the lamp, transformers may cause drift in the setting.
- v) Suitable for use in dark room (conditions apply).

1.5 ELECTRICAL COMPARATOR

In this comparator, the movement of the measuring contact (plunger) is converted into an electrical signal. This electrical signal is recorded by an instrument which can be calibrated in terms of plunger movement.

It consists of an armature suspended on thin steel strips. This armature is suspended between two coils A and B. The distance between armature and the two coils are equal. The coils are connected in two arms of a Wheatstone bridge. A galvanometer is also connected in the Wheatstone bridge circuit.

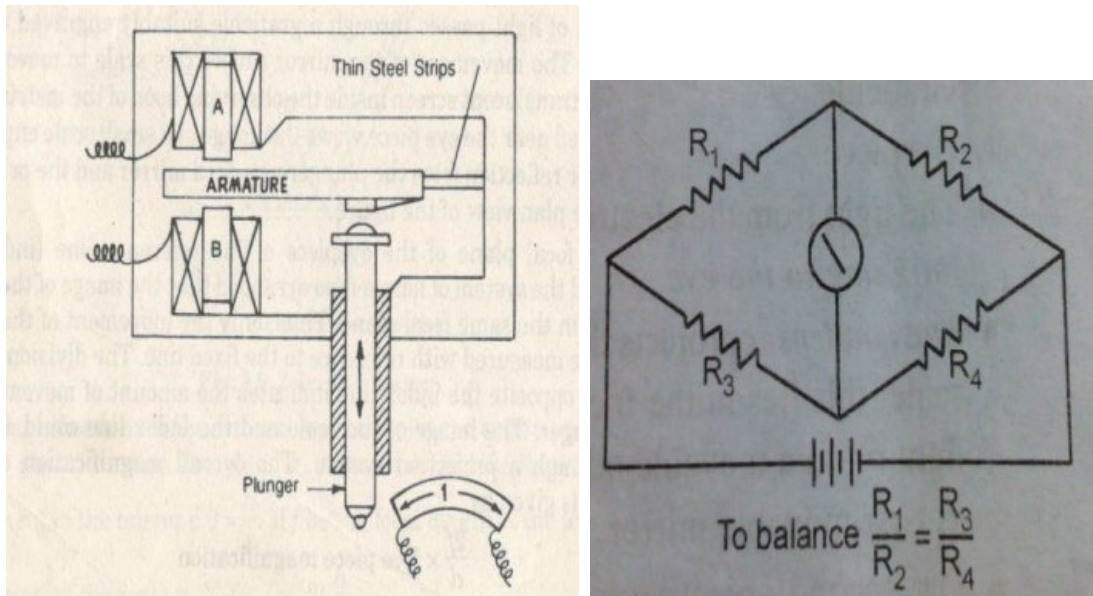


Figure 1.6 Electrical Comparator

When the object is in correct dimension the Wheatstone bridge is balanced and no current flows through the galvanometer. Any variation in the dimension of the object will move the measuring plunger lightly. This changes the position of armature. Now the Wheatstone bridge unbalances and causes a current to pass through the galvanometer. This current is directly proportional to the variation of the object from the standard. Since the galvanometer readings are calibrated in terms of plunger movement, we can directly take the readings. This type of comparator gives magnification of 30,000 times.

1.5.1 ADVANTAGES OF ELECTRICAL COMPARATOR

- i) Less number of moving parts.
- ii) High magnification.
- iii) Easy to handle.
- iv) Non sensitive to vibrations.
- v) Small and compact.
- vi) Less error.
- vii) Indicating instrument can be placed away from the measuring unit.

1.5.2 DISADVANTAGES OF ELECTRICAL COMPARATOR

- i) Depends on external electrical supply.
- ii) Fluctuations in voltage/frequency may affect readings.
- iii) Heating of coils cause zero drift.
- iv) Higher cost compared to mechanical comparators.
- v) Poor reliability if measuring unit is away from indicating unit.

1.6 ELECTRONIC COMPARATOR

It consists of a probe tip, oscillator, amplifier, de-modulator and a dial. The movement at the probe tip actuates an inductance transducer, which is supplied with an alternating current from the oscillator. The transducer converts this movement into an electrical signal which is then amplified and fed via an oscillator to the de-modulator. The current in DC form, then passes to the meter and the probe tip movement is displayed as a linear measurement.

Various measuring and control units can be added to provide an extremely wide range of single or multiple measurements simultaneously.

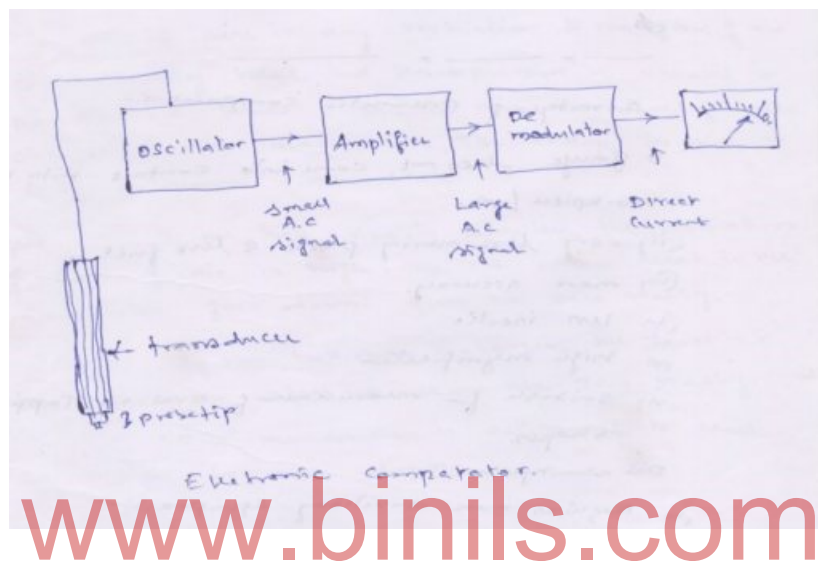


Figure 1.7 Electronic Comparator

1.7 PNEUMATIC COMPARATOR

It has a water tank fitted with manometer. A dip tube is dipped into water. A restriction chamber is connected at the top of the dip tube. The restriction chamber is connected to a flexible pipe through a control jet. The top of the manometer is also connected to the flexible tube. Compressed air enters the dip tube through the restriction chamber. The air expands in the tube and maintains a constant head of water. The excess air will escape as air bubbles. But air from the suction chamber flows through flexible tube and escapes out through the measuring head. Now the level of the water both in the tank and manometer will be the same.

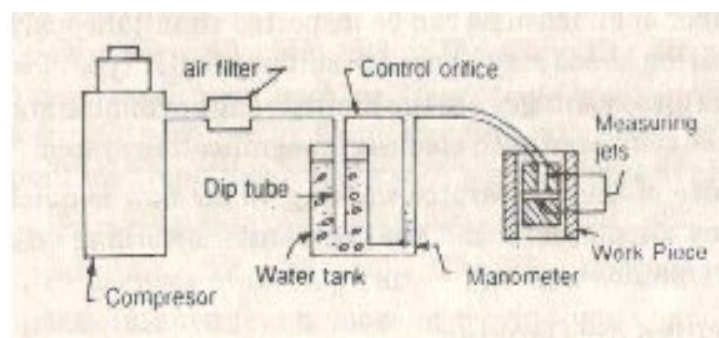


Figure 1.8 Pneumatic comparator

If there is any restriction to the flow of air in measuring head due to the work piece, a back pressure is created in flexible tube. Due to this, the level of water changes in the manometer. The change in the level is shown in the scale. If the hole size is smaller, the restriction to the flow of air is high. So the water level in the manometer goes down from the zero reading. If the hole size is larger, the level in the manometer rises up above the zero reading. The manometer is calibrated to read the changes in size directly

1.7.1 ADVANTAGES OF PNEUMATIC COMPARATOR

- i) Gauge does not come into contact with the work piece / job.
- ii) Very less moving parts & less friction.
- iii) More accuracy.
- iv) Less inertia.
- v) High magnification.
- vi) Suitable for measurement of oval and taper shapes.

1.7.2 DISADVANTAGES OF PNEUMATIC COMPARATOR

- i) Requires more auxiliary equipments.
- ii) Scale is not uniform.
- iii) Not easily portable.
- iv) Different gauge heads are needed for different dimensions.

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REVIEW QUESTIONS

PART -A

1. What is Comparator?
2. Define Mechanical Comparator.
3. What is an Electrical Comparator?
4. What is Optical Comparator?
5. What is Pneumatic Comparator?
6. List the parts of Dial Gauge.
7. Compare electrical and pneumatic comparator.

PART - B

1. State the advantages of mechanical comparator.
2. Differentiate comparator and measuring instrument.
3. State the disadvantages of mechanical comparator.
4. State the advantages of Optical Comparator.
5. State the disadvantages of Optical Comparator.

6. State the advantages of Electrical Comparator.
7. State the disadvantages of Electrical Comparator.
8. State the advantages of Pneumatic Comparator.
9. State the disadvantages of Pneumatic Comparator.

PART – C

1. Explain the construction and working principle of Dial Gauge.
2. Explain with suitable diagram the working of Reed Type Comparator.
3. Explain the construction and working of Electrical Comparator.
4. Explain the working of electronic comparator with a block diagram.
5. Explain the construction and working principle of Zeiss Ultra Optimeter.
6. Explain with suitable diagram the working of Optical Lever Comparator.
7. Explain the construction and working principle of Pneumatic Comparator.

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UNIT II - MEASUREMENT OF VELOCITY AND ACCELERATION

Linear Velocity Measurement

Doppler Effect Method

Linear Encoder

Angular Velocity Measurement

Tachometer

Eddy Current or Drag Cup Rotor AC Tachogenerator

Angular Encoder

Accelerometer

Seismic Accelerometer

Piezo electric Accelerometer

Strain Gauge Accelerometer

Principle of Operation, Construction, Advantages and Disadvantages of the above

2.1 Introduction:

The velocity of an object is the rate of change of displacement with respect to time. The velocity is equivalent to the specification of speed and direction of motion. So the velocity is the speed of something in a given direction.

The scalar absolute value (magnitude) of velocity is called speed, being a derived unit whose quantity is measured in SI system as metres per second. For example, 5metres per second is a scalar, whereas 5metres per second to the east is a vector.

2.2 LINEAR VELOCITY MEASUREMENT

The linear velocity of an object is the rate of change of displacement in a particular direction. It is a vector quantity and has both direction and magnitude. The various methods of linear velocity measurement are

1. Doppler Effect Method
2. Linear Encoder Method

2.2.1 Doppler Effect Method

The Doppler Effect or Doppler Shift is the apparent change in the frequency of the waves occurring when the source and observer are in motion relative to each other.

This phenomenon is applicable to waves in general like, sound, light and microwaves. It is first observed for sound waves and is named after the Austrian Physicist and Mathematician Christian Doppler, who proposed the phenomenon in 1842. This phenomenon was illustrated by having people to listen to the pitch of the whistle from the oncoming train. The high pitched whistle would gradually change to a lower pitch whistle as the train passes the observer.

The frequency of the sound waves will increase when the source and the observer approach each other and the frequency decreases when they move apart.

The Doppler Radar is used for measurement of linear velocity of the moving objects. It is similar to the one, commonly used by the traffic police to determine the speed of the over speeding vehicles in the roads and highways. It consists of two ultrasonic transducers, one act as transmitter and the other acts as receiver. The ultrasonic beam from transmitter is pointed on a moving object and a reflected beam will be received from the moving object in the receiver. When the object is in motion, the frequency of the reflected signals differs from the transmitted signal. This difference in frequency of the ultrasonic wave is used to measure the velocity of the moving object.

- a. If the object is moving towards the radar unit, the frequency of the reflected signal received will be greater than the transmitted signal frequency.

$$f_r = f_t + f_d$$

- b. If the object is moving away from the radar unit, the frequency of the reflected signal received will be less than the transmitted signal frequency.

$$f_r = f_t - f_d$$

where, f_t = frequency of transmitted signal

f_r = Frequency of reflected signal

f_d = Doppler Shift frequency

2.2.2 Linear Encoder

In general, encoder is a device that converts information from one format or code to another format or code for the purpose of standardization.

In the measurement of velocity, encoder is a transducer used to translate the rotary or linear motion into a digital signal. The encoders may be incremental or absolute.

The linear encoders are used in metrology instruments, CNC machines, Digital Calipers, Coordinate Measuring Machines (CMM), etc. to determine the linear displacement. The linear encoder technologies include resistive, optical, magnetic, inductive or capacitive methods.

Contact or Brush type (Resistive) Encoders

The resistive encoder consists of stationary brush type contacts and coded movable strip. The shaded areas in the strip are made of conducting material and the unshaded areas are made of insulating material. In the model shown in the figure, the code pattern used is the familiar binary system and many commercial encoders use different code patterns such as Gray code, etc.

The brushes are placed on the strip, which act as sliding contacts. The circuits of the sliding contacts, which come in contact with the conducting areas are completed and the circuits of the others which make contacts with insulated area are not completed. The read out lamps are shown for explanatory function. Thus the encoder gives out a digital read-out which is an indication of position and hence the encoder determines the

displacement. The table shows the digital numbers corresponding to the different brush positions.

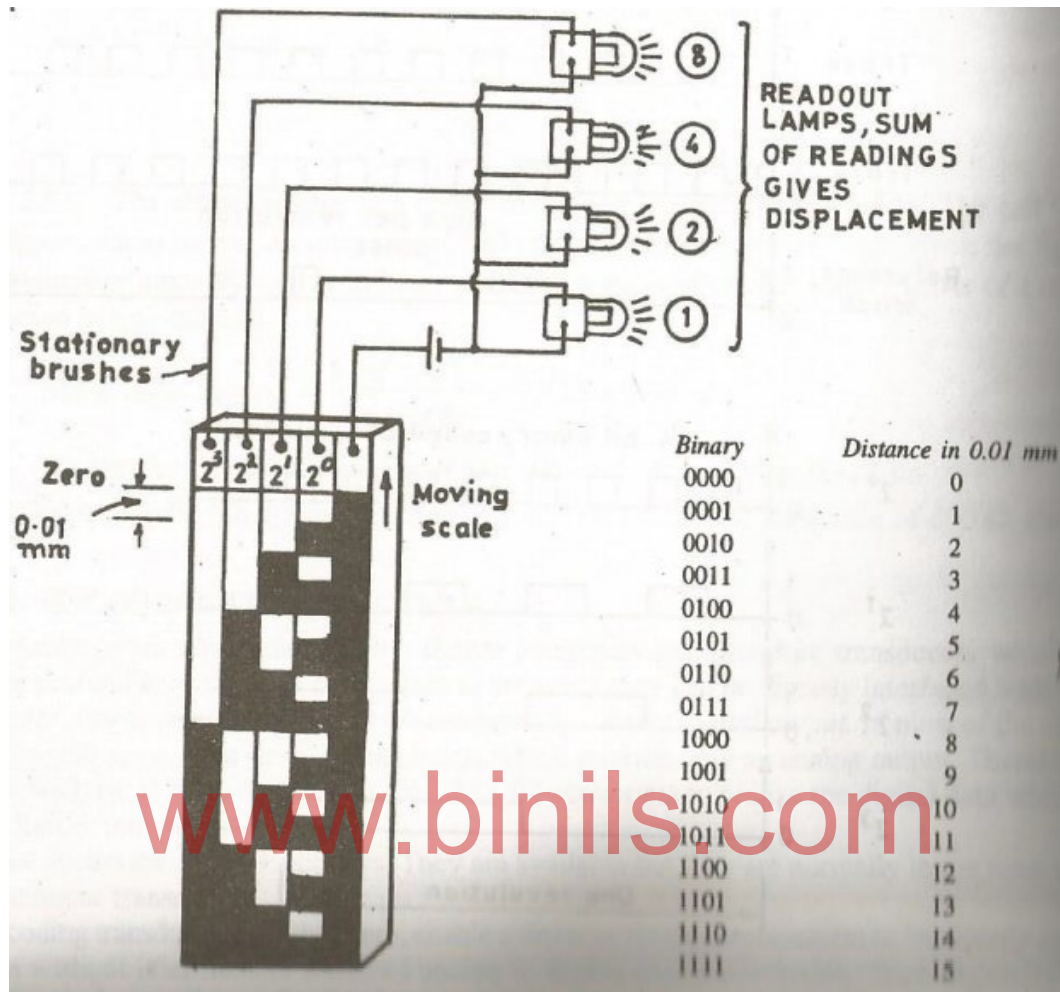


Fig 2.1 Contact Type Linear or Translational Encoder

Advantages

1. It is relatively inexpensive compared to other encoders.
2. It can be made to any degree of desired accuracy.

Disadvantages

1. The major problem is the wear of contacts and the resulting maintenance problem.

2.3 Angular Velocity Measurement

The angular velocity is defined as the rate of change of angular displacement and it is a measure of how fast the object is turning.

A Tachometer is an instrument for measuring the rotational speed of a motor shaft or any other machine. It usually displays the number of revolution during the period of contact or by indicating the number of revolutions per minute (RPM) on a calibrated dial or digital display.

The tachometers are classified as :

1. Mechanical Tachometers
2. Electrical Tachometers
 - a. Eddy current or Drag Cup Rotor AC Tachogenerator

2.3.1 Eddy Current or Drag Cup Rotor AC Tachogenerator

In an eddy current or drag type tachometer, the test shaft rotates a permanent magnet and this induces eddy currents in a drag cup or disc held close to the magnet. The eddy currents produce a torque which rotates the cup against the torque of a spiral spring. The disc turns in the direction of the rotating magnetic field until the torque developed equals that of the spring. A pointer attached to the cup indicates the rotational speed on a calibrated scale. The automobile speedometers operate on this principle and measure the angular speed of the wheels. The rotational measurement is subsequently converted into linear measurement by assuming some average diameter of the wheel, and the scale is directly calibrated in linear speed units.

The Eddy current tachometers are used for measuring rotational speeds upto 12,000 rpm with an accuracy of $\pm 3\%$.

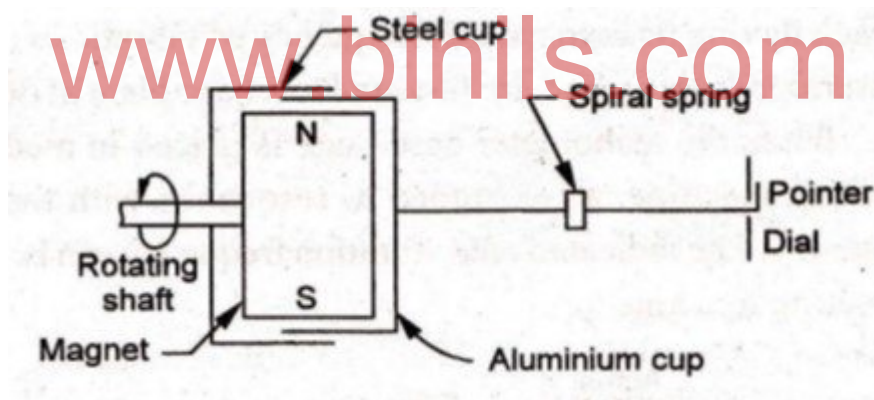


Fig.2.2 Drag Cup Tachometer

2.3.2 Tachogenerators

These tachometers employ small magnet type d.c or a.c generators which translate the rotational speeds into d.c or a.c voltage signal. The operating principle of such tachometers is illustrated in Fig. Relative perpendicular motion between a magnetic field and conductor results in voltage generation in the conductor.

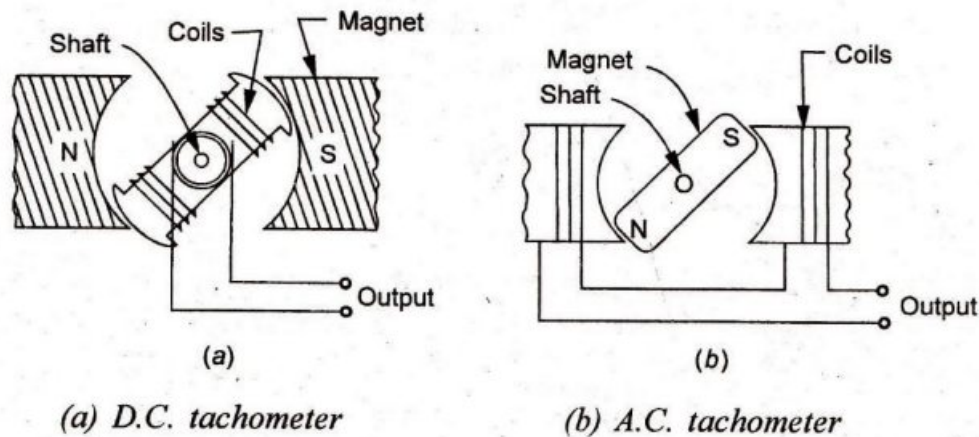


Fig. 2.3 DC & AC Tachometer

(i) D. C. Tachometer Generator:

This is an accurately made dc. generator with a permanent magnet of horse-shoe type. With rotation of the shaft, a pulsating dc. Voltage proportional to the shaft speed is produced, and measured with the help of a moving coil voltmeter having uniform scale and calibrated directly in terms of speed. The tachometer is sensitive to the direction of rotation and thus can be used to indicate this direction by the use of an indicator with its zero point at mid-scale. For greater accuracy, air gap of the magnetic paths must be maintained as uniform as possible. Further, the instrument requires some form of commutation which presents the problem of brush maintenance.

(ii) A.C. Tachometer Generator:

The unit embodies a stator surrounding a rotating permanent magnet. The stator consists of a multiple pole piece (generally four), and the permanent magnet is installed in the shaft whose speed is being measured. When the magnet rotates, an a.c. voltage is induced in the stator coil. The output voltage is rectified and measured with a permanent magnet moving coil instrument. The instrument can also be used to measure a difference in speed of two sources by differentially connecting the stator coils.

Tachogenerators have been successfully employed for continuous measurement of speeds upto 500 rpm with an accuracy of $\pm 1\%$.

2.3.3 Angular Encoder or Shaft Angle Encoder

A Shaft encoder is a digital device that converts the angular position of the shaft to a digital code.

The key components of a rotary encoder are the disc, light sources and detectors, and electronics. The disc contains a unique pattern of concentric etched circles and alternates between opaque and transparent segments. This pattern provides unique bit configurations and is used to assign specific positions. For every concentric ring in a rotary encoder, there is a light source and light detector which identify lines etched on the disk. The electronics consist of an output device which takes the signal obtained from the sensor (light/detector source) to provide feedback of position and/or velocity. All of these components are enclosed in a single housing unit which is mounted on the shaft of a motor or machine.

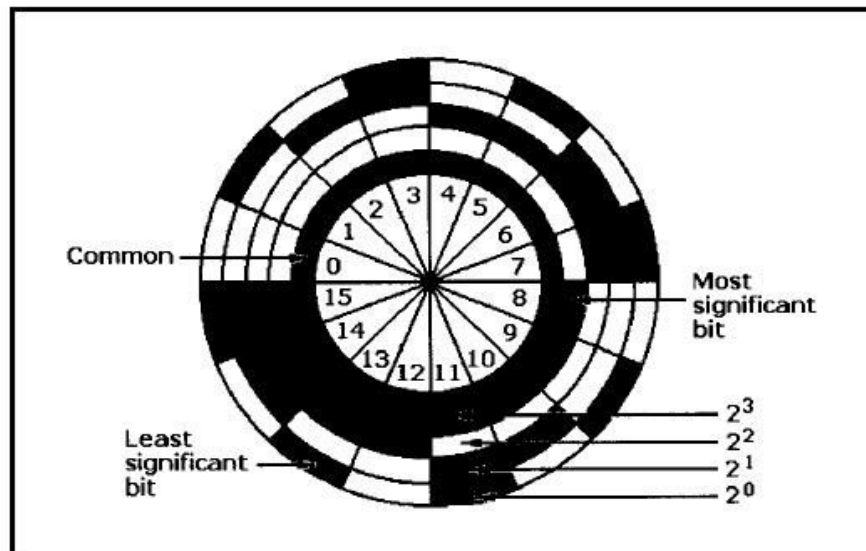


Fig.2.4 Binary Code Disc

The above figure shows a binary code disc for an optical shaft encoder. The disc is divided into circular tracks and each of these is divided into segments in a manner depending upon the code being used. The black or opaque sectors represent a binary value of 1 and the white or transparent sectors represent binary 0. This four bit binary code disk can count from 0 to 15.

If electrical methods are used for detection, the segments are made conducting and non-conducting alternatively.

Thus if the scale is scanned radially, a binary 0 or 1 is obtained from each track depending upon the angular position of the disc. The accuracy depends upon the number of tracks in the disc. If there are n tracks, the accuracy will be $360^\circ \div 2^n$.

For the four track disc, the resolution or accuracy is $360^\circ \div 2^4 = 22.5^\circ$

For the ten track disc, the resolution or accuracy is $360^\circ \div 2^{10} = 0.3515^\circ$.

2.4 ACCELEROMETER

An accelerometer is an electromechanical device that will measure either static or dynamic forces of acceleration. The static forces include gravity, like the constant force of gravity pulling at your feet, while dynamic forces include vibrations and movement.

By measuring the amount of static acceleration due to gravity, you can find out the angle the device is tilted with respect to the earth.

By sensing the amount of dynamic acceleration, you can analyze the way the device is moving or vibrating.

Accelerometers can be used to measure vibration on vehicles, machines, buildings, process control systems and safety installations. They can also be used to measure seismic activity, inclination, machine vibration, dynamic distance and speed with or without the influence of gravity.

2.4.1 Seismic Accelerometer:

In a seismic accelerometer the displacement of a mass resulting from an applied force is measured and correlated to the acceleration. The figure shows the schematics of a common spring mass damper system which accomplishes this task. The mass is supported by a spring and damper is connected to the housing frame. The housing frame is rigidly attached to the machine whose acceleration characteristics are to be determined. When acceleration is imparted by the machine to the housing frame, the mass moves relative to the frame, and this relative displacement between the mass and frame is sensed and indicated by an electrical displacement transducer. This displacement of the mass is proportional to the acceleration. Hence a measure of displacement of the mass becomes a measure of acceleration.

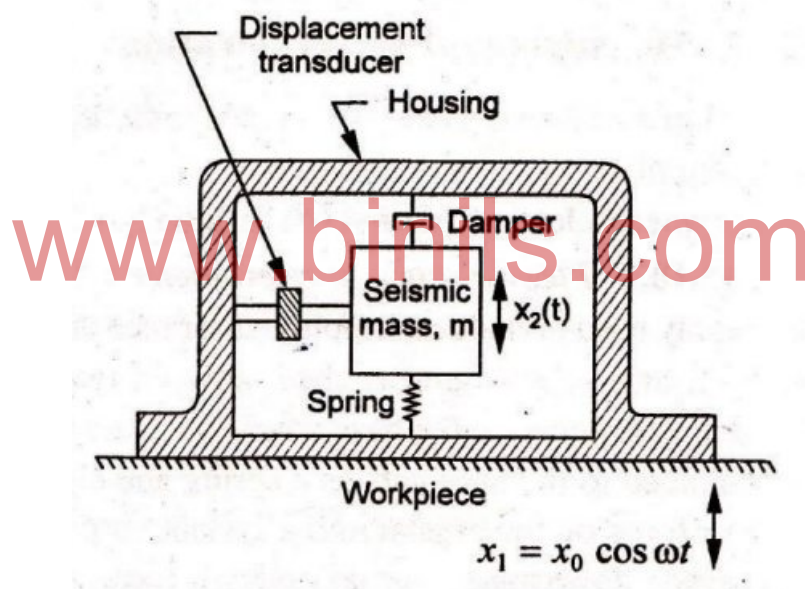


Fig.2.5 Seismic Accelerometer

2.4.2 PIEZO-ELECTRIC ACCELEROMETER:

The unit is perhaps the simplest and most commonly used transducer employed for measuring acceleration. The sensor consists of a piezo-electric crystal sandwiched between two electrodes and has a mass placed on it. The unit is fastened to the base whose acceleration characteristics are to be obtained. The can threaded to the base acts as a 'spring and squeezes the mass against the crystal. Mass exerts a force on the crystal and a certain output voltage is generated. If the base is now accelerated downward, inertial reaction force on the base acts upward against the top of the can. This relieves stress on the crystal.

From Newton's second law,

$$\text{Force} = \text{mass} \times \text{acceleration}$$

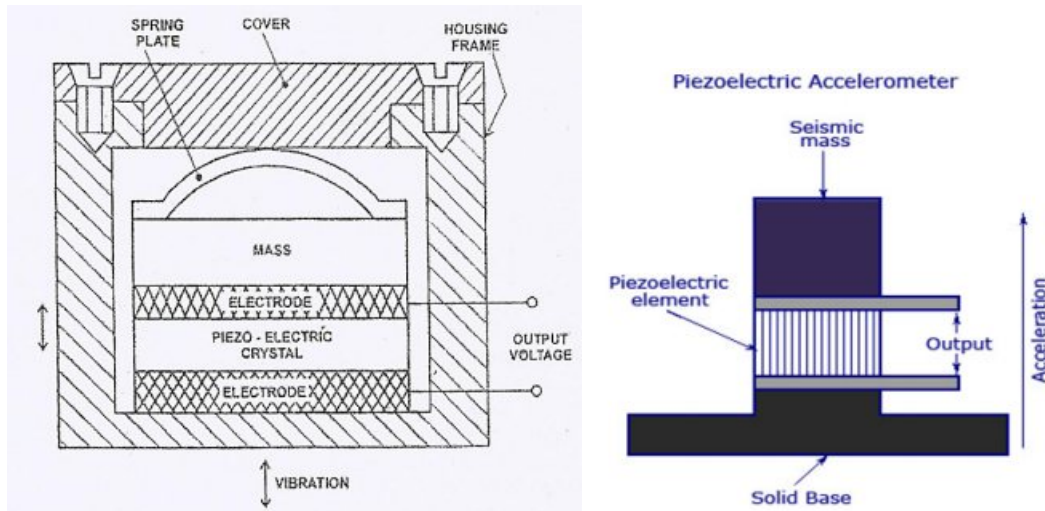


Fig.2.6 Piezo-electric Accelerometer

Advantages

1. The instrument is small in size and less weight.
2. Rugged and inexpensive device
3. High output impedance.
4. High frequency response from 10 Hz to 50 kHz
5. High sensitivity varies from 10 to 100 mv/g, where $g = 9.807 \text{ m/s}^2$
6. Capability to measure acceleration from a fraction of g to thousands of g.
7. Used to measure vibration and shock.

Limitations

1. Sensitive to changes in temperature
2. Subject to hysteresis errors.

2.4.3 Strain Gauge Accelerometer

In the Strain Gauge Accelerometer, the electrical strain gauges are used for displacement measurement as shown in the figure. The seismic mass is mounted on a cantilever beam. A strain gauge is mounted on each side of the cantilever beam in order to sense the strain in the beam resulting from the vibrational displacement of the mass. The damping is provided by completely filling the housing with a viscous fluid.

The output of the strain gauge is connected to an appropriate Wheatstone Bridge circuit, whose output indicates the relative displacement of the mass with respect to the housing frame.

The large length of the cantilever beam is used to accommodate the strain gauges. This results in low natural frequencies.

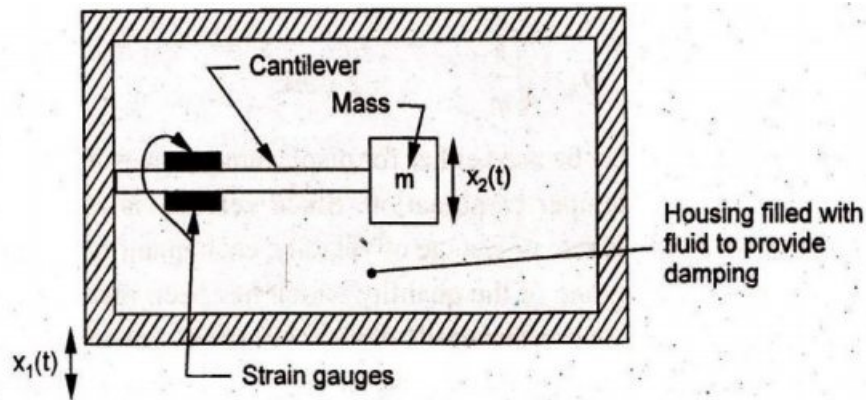


Fig.2.7 Strain Gauge Accelerometer

The primary advantages of the Strain Gauge accelerometer over the piezoelectric accelerometer are that they are more sensitive and can be used for measurement of small and static accelerations.

Review Questions:

Part A (2 marks)

1. Define Linear Velocity and give its unit.
2. Define Angular Velocity and give its unit.
3. What is a Tachometer?
4. What is an Encoder?
5. What is the use of an Accelerometer?

Part B (3marks)

1. Brief Doppler Effect.
2. What is an Angular Encoder?
3. Draw the diagram of Drag Cup Rotor Tachometer.
4. Give the construction of Seismic Accelerometer.

Part C (10 marks)

1. Explain any one method of linear velocity measurement.
2. Explain the Doppler Effect method of linear velocity measurement.
3. Explain the construction and working of Drag Cup Rotor Tachometer.
4. Explain the construction and working of Angular Encoder.
5. Explain the construction and working of Piezo-electric Accelerometer and give its advantages.

Unit III - MEASUREMENT OF FORCE, TORQUE AND SHAFT POWER

FORCE MEASUREMENT

Definition

Principle of Operation and Construction of

Equal and Unequal Arm Balance

Pendulum Scale

Elastic Element

Springs

Proving Ring

Load Cell

Hydraulic Load Cell

Pneumatic Load Cell

Strain Gauge Load Cell

TORQUE MEASUREMENT

Definition

Principle of Operation and Construction of

Gravity Balance Method

Optical Torsion Method

Electrical Torsion Method

Strain Gauge Torsion Method

SHAFT POWER MEASUREMENT

Prony Brake Dynamometer

Rope Brake Dynamometer

Fluid Friction (Hydraulic) Dynamometer

Eddy Current Dynamometer

DC Dynamometer

3.1 FORCE MEASUREMENT

Force represents the mechanical quantity which changes the relative motion or shape of the body to which it is applied.

Force can be a push or a pull. It is not something you can see or touch, but can see it in action. Forces can be measured using a device called force meter.

A force has both magnitude and direction, making it a vector quantity. It is measured in the SI unit of Newton ($kg.m/sec^2$) and represented by the symbol F.

The original form of Newton's second law states that the net force acting upon an object is equal to the rate at which its momentum changes with time. If the mass of the object is constant, this law implies that the acceleration of an object is directly proportional to the net force acting on the object,

$$\text{ie., } F = m \times a \text{ Newtons}$$

where m = mass in kg, and a = acceleration in m/s^2 .

3.1.1 BASIC METHODS OF MEASUREMENT OF FORCE

The unknown forces may be measured by the following methods:

1. Balancing the unknown force against the known gravitational on a standard mass.(eg.: scales and balances)
2. Applying a force to an elastic member and then measuring the resulting deflection in terms of force (eg.: proving ring)
3. Translating the force to a fluid pressure and then measuring the resulting pressure (eg.: hydraulic and pneumatic load cells)
4. Applying the force to a known mass and then measuring the resulting acceleration.

3.2 Scales and Balances

A weighing machine is commercially known as Scale or Balance, and is used for measurement of force and torque by comparison of weights.

Force or weight is indicated by making a comparison between the forces due to gravity acting on a standard mass and the force due to gravity acting on unknown mass.

It is based on the principle of equilibrium produced by two torques, as a result of forces acting at equal or different distances from the fulcrum.

3.2.1 EQUAL ARM BALANCE

The most simple weight or force measuring system is the ordinary equal arm balance as shown in the figure. The device operates on the principle of moment comparison.

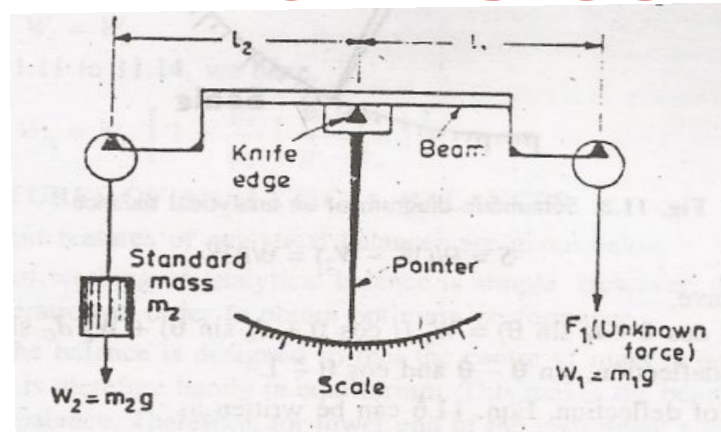


Fig.3.1 EQUAL ARM BALANCE

It consists of a beam pivoted on a knife edge fulcrum placed exactly at the centre of the beam. A pointer is attached to the centre of the beam, which points vertically downwards, when the beam is in equilibrium.

The unknown force is applied to one side of the beam and the torque (rotating moment) produced due to this force is balanced by standard masses on the other side of the beam.

The unknown force F_1 produces a moment $F_1 l_1$ and this moment is matched by a moment produced by a standard mass m_2 . The latter moment is $m_2 g l_2$.

For equilibrium, $F_1 l_1 = m_2 g l_2$,

Therefore the unknown force $F_1 = m_2 g l_2 / l_1$,

In case the force F_1 is exerted by a weight W_1 , we have

$$W_1 = m_2 g l_2 / l_1 \\ = W_2 l_2 / l_1, \text{ where weight } W_2 = m_2 g$$

For an equal arm analytical balance, $l_1 = l_2$ and therefore $W_1 = W_2 = m_2 g$.

Therefore an equal arm balance may be used for measuring a force against a known mass. This balance may be used for measuring weight of equal magnitude with good accuracy, since the gravitational force acting on the two masses is equal for any location.

3.2.2 UNEQUAL ARM BALANCE

The unequal arm analytical balance suffers from the major disadvantage that it requires a set of weights which are atleast as heavy as the heaviest weight to be measured.

For weighing of heavier objects, with the help of lighter weights, the balances with beams having arms of unequal lengths are designed. The unequal arm balance uses two arms. One is called the load arm and the other is called the power arm. The load arm is associated with the load, ie., the weight force to be measured, while the power arm is associated with power, ie., the force produced by the counterpoising weights required to set the balance in equilibrium.

The balance is specified in two ways:

1. In terms of mechanical advantage (MA), which is the ratio of load to power,

$$MA = \frac{\text{Load}}{\text{Power}}$$

2. In terms of multiple M which is defined as

$$MA = \frac{\text{Power Arm}}{\text{Load Arm}} \\ = \frac{\text{distance between fulcrum and power point}}{\text{distance between fulcrum and load point}}$$

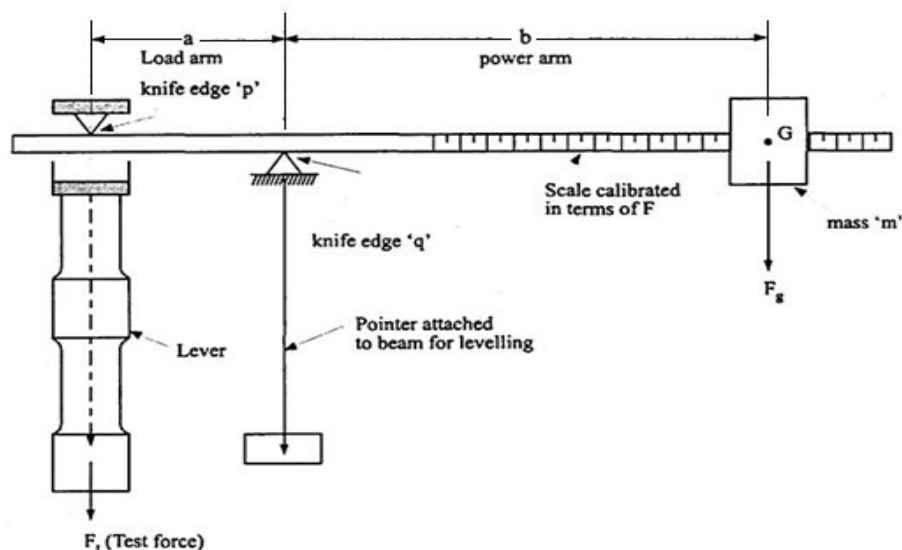


Fig.3.2 Unequal Arm Balance

The figure shows the typical arrangement of unequal arm balance. The mass, 'm' acts as power on the beam and exerts a force of F_g due to gravity where $F_g = m \times g$. This force acts as counterpoising force against the load which may be a test force F_t .

The beam is pivoted on a knife edge 'q'. The test force F_t is applied by a screw or a lever through a knife edge 'p' until the pointer indicates that the beam is horizontal. For balance of moments,

$$\begin{aligned} F_t \times a &= F_g \times b \text{ or} \\ \text{Test Force } F_t &= F_g \times b/a \\ &= m \times g \times b/a \\ &= \text{constant} \times b \text{ (provided that } g \text{ is constant)} \end{aligned}$$

Therefore the test force is proportional to the distance 'b' of the mass from the pivot. Hence, if mass 'm' is constant and the test force is applied at a fixed distance 'a' from the knife edge 'q' (i.e., the load arm is constant), the right hand of the beam (i.e., the power arm) may be calibrated in terms of force F_t . If the scale is used in different gravitational fields, a correction may be made for change in value of 'g'. The set-up shown in Fig. is used for measurement of tensile force. With suitable modifications, it can be used for compression, shearing and bending forces. This machine can also be used for the measurement of unknown mass.

Suppose force F_t is produced by an unknown mass m_t .

Then, $F_t = m_t g$

Hence, for balance, $m_t \times g \times a = m \times g \times b$

Or $m_t = m \times b/a$
 $= \text{a constant} \times b$

Therefore, the power arm 'b' may be calibrated to read the unknown mass m_t directly, if 'm' and 'a' are fixed. This forms the basis of countless weighing (i.e., mass measuring) machine.

3.2.3 PENDULUM SCALE

The pendulum scale is a deflection-type instrument. It is a moment comparison device. The unknown force is converted to torque which is then balanced by the torque of a fixed standard mass arranged as a pendulum.

The practical version of this principle utilizes specially shaped sectors and steel tapes to linearize the inherently nonlinear torque-angle relation of a pendulum.

Description:

The main parts of a pendulum scale are as follows:

The scale's frame carries support ribbons. These support ribbons are attached to the sectors as shown in the figure.

The loading ribbons are attached to the sectors and load rod as shown in the figure. The load rod in turn is attached to the weighing platform.

The two sectors are connected on either side of the equalizer beam. The sectors carry counter weights.

To the center of the equalizer beam is attached a rack and pinion arrangement.

A pointer is attached to the pinion which sweeps over a calibrated scale.

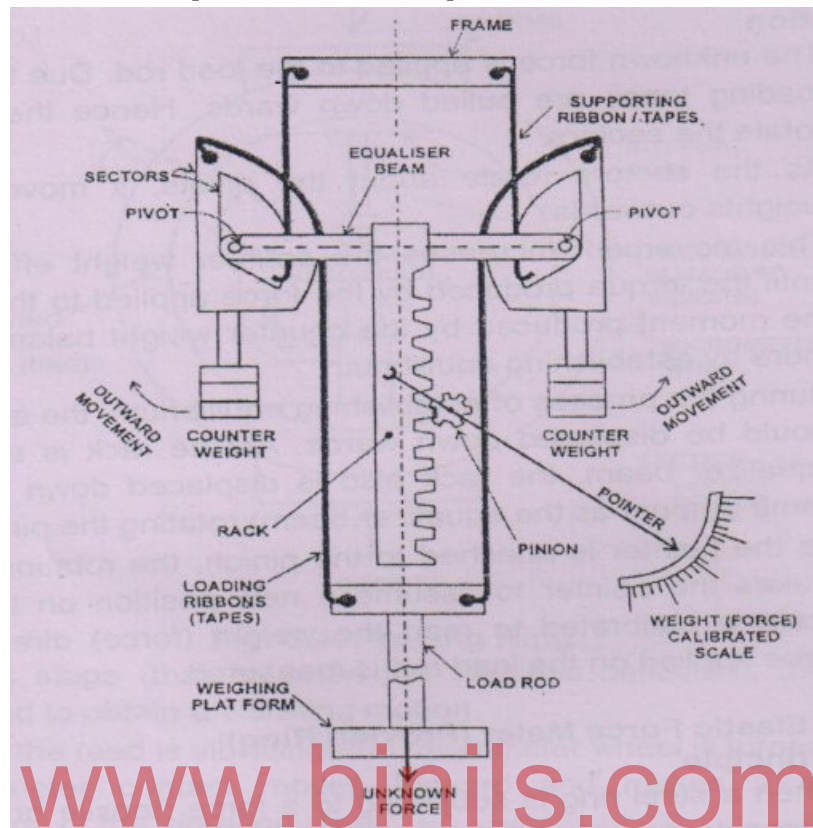


Fig.3.3 Pendulum Scale

Operation:

The unknown force is applied to the load rod. Due to this force, the loading tapes are pulled downwards. Hence the loading tapes rotate the sectors.

As the sectors rotate about the pivots, it moves the counter weights outwards, This movements increases the counter weight effective moment until the torque produced by the force applied to the load rod and the moment produced by the counter weight balance each other, thereby establishing an equilibrium.

During the process of establishing equilibrium, the equalizer beam would be displaced downwards. As the rack is attached to the equalizer beam, the rack also is displaced downwards rotating the pinion.

As the pointer is attached to the pinion, the rotation of the pinion makes the pointer to assume a new position on the scale. The scale is calibrated to read the force directly. Thus the force applied on the load rod is measured.

3.3 Elastic Elements

These devices measure the force by applying it to an elastic member and then measuring the elastic deformation or deflection. Within the elastic limit of the materials, the deflection of the element is proportional to the force.

3.3.1 Spring

A **spring** is an elastic object used to store mechanical energy. Springs are usually made out of spring steel. There are a large number of spring designs; in everyday usage the term often refers to coil springs.

When a spring is compressed or stretched from its resting position, it exerts an opposing force approximately proportional to its change in dimension. The spring constant of a spring is the change in the force it exerts, divided by the change in deflection of the spring.

A spring balance is an example where a force may be converted to a displacement based on the spring constant. For a spring element the relationship between force F and displacement ' x ' is linear and is given by $F = K x$, where K is the spring constant.

Simplest device of this type is in fact the spring balance whose schematic is shown in Figure. The spring is fixed at one end and at the other end hangs a pan. The object to be weighed is placed in the pan and the position of the needle along the graduated scale gives the weight of the object.

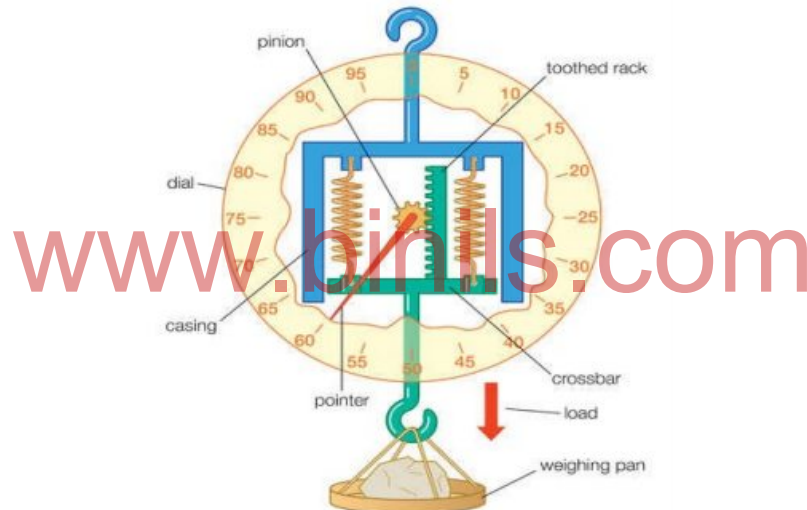


Fig 3.4 Spring Balance

3.3.2 PROVING RING

The proving ring is a device used to measure force. It consists of an elastic ring of known diameter with a measuring device located in the center of the ring.

Proving rings come in a variety of sizes. They are made of a steel alloy. Manufacturing consists of rough machining from annealed forgings, heat treatment, and precision grinding to final size and finish.

Proving rings have evolved over time; however, they are still manufactured according to design specifications established in 1946 by the National Bureau of Standards (NBS), the predecessor of the National Institute of Standards and Technology (NIST). Those specifications can be found in the Circular of the National Bureau of Standards C454, issued in 1946. The concept behind the proving ring is illustrated in the diagram below.

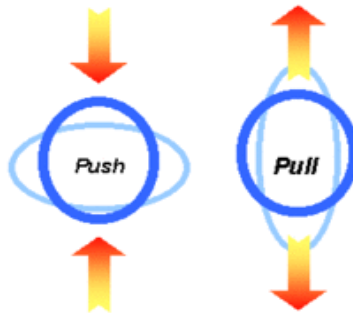


Fig 3.5 Proving Ring – Working Principle

Proving rings can be designed to measure either compression or tension forces. Some are designed to measure both. The basic operation of the proving ring in tension is the same as in compression. However, tension rings are provided with threaded bosses and supplied with pulling rods which are screwed onto the bosses.

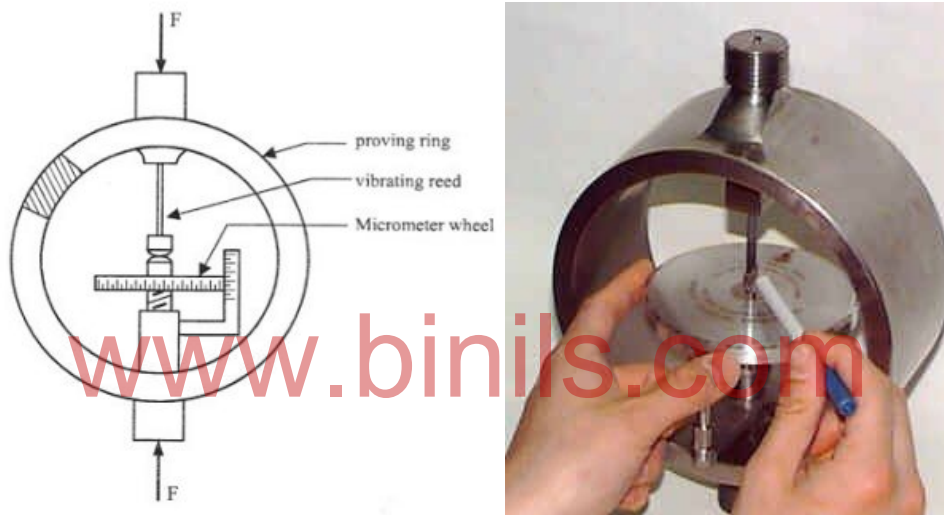


Fig 3.6 Proving Rings

The proving ring consists of two main elements, the ring itself and the diameter-measuring system. Forces are applied to the ring through the external bosses. The resulting change in diameter, referred to as the deflection of the ring, is measured with a micrometer screw and the vibrating reed mounted diametrically within the ring.

The micrometer screw and the vibrating reed are attached to the internal bosses of the ring. In modern rings, the upper and lower internal and external bosses are machined as an integral part of the ring to avoid mechanical interferences during the application of the force.

To read the diameter of the ring, the vibrating reed is set in motion by gently tapping it with a pencil. As the reed is vibrating, the micrometer screw on the spindle is adjusted until the button on the spindle just contacts the vibrating reed, dampening out its vibrations. When this occurs, a characteristic buzzing sound is produced. At this point a reading of the micrometer dial indicates the diameter of the ring.

The number of divisions on the micrometer dial and the graduation of the vernier index vary by type of proving ring. Typically, proving rings are designed to have

a deflection of about 0.84 mm (0.033 in) to 4.24 mm (0.167 in). The relative measurement uncertainty can vary from 0.075 % to about 0.0125 %.

3.4 LOAD CELL

A load cell is a transducer that is used to create an electrical signal whose magnitude is directly proportional to the force being measured. The various types of load cells include **hydraulic load cells, pneumatic load cells and strain gauge load cells.**

3.4.1 STRAIN GAUGE LOAD CELL

A Load cell is a transducer that is used to convert a force into an electrical signal. This conversion is indirect and happens in two stages. Through a mechanical arrangement, the force being sensed deforms a strain gauge. The strain gauge measures the deformation (strain) as an electrical signal, because the strain changes the effective electrical resistance of the wire. A load cell usually consists of four strain gauges in a Wheatstone bridge configuration. Load cells of one strain gauge (Quarter Bridge) or two strain gauges (half bridge) are also available. The electrical signal output is typically in the order of a few mill volts and requires amplification by an instrumentation amplifier before it can be used. The output of the transducer can be scaled to calculate the force applied to the transducer.

Basic Principle of Strain Gauge Load Cell

When steel cylinder is subjected to a force, it tends to change in dimension. On this cylinder, if the strain gauges are bonded, the strain gauge also is stretched or compressed, causing a change in its length and diameter. This change in dimension of the strain gauge causes its resistance to change. This change in resistance or output voltage of the strain gauge becomes a measure of applied force.

Load cells are used for quick and precise measurements. Compared with other sensors, load cells are relatively more affordable and have a longer life span.

Construction of Strain Gauge Load cell

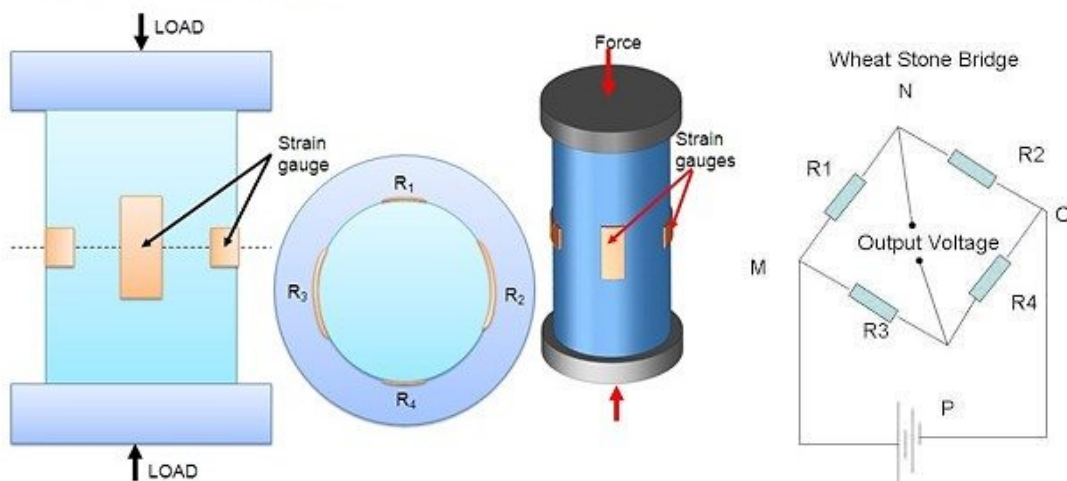


Fig.3.7 Strain Gauge Load Cell

The main parts of the strain gauge load cell are as follows. There is a cylinder made up of steel on which four identical strain gauge are mounted and out of four strain gauges, two of them (R1 and R4) are mounted along the direction of the applied load (vertical gauges). The other two strain gauges (R2 and R3 Horizontal gauges) are mounted circumferentially at right angles to gauges R1 and R4.

Operation of Strain Gauge Load cell

Let study the operation in two cases

Case 1

When there is no load (force) on the steel cylinder, all the four gauges will have the same resistance. As the terminals N and P are at the same potential, the wheat stone bridge is balanced and hence the output voltage will be zero.

Case 2

Now the load (force) to be measured (say compression force) is applied on the steel cylinder. Due to this, the vertical gauges R1 and R4 will undergo compression and hence there will be a decrease in resistance. At the same time, the horizontal gauges R2 and R3 will undergo tension and there will be an increase in resistance. Thus when strained, the resistance of the various gauges change.

Now the terminal N and P will be at different potential and the change in output voltage due to the applied load (force) becomes a measure of the applied load force when calibrated.

Uses of Strain Gauge Load Cell

Strain gauge load cells are used when the load is not steady.

Strain gauge load cells are used in vehicle weigh bridges, and tool force dynamometers.

3.4.2 HYDRAULIC LOAD CELL

Basic Principle of Hydraulic Load Cell

When a force is applied on a liquid medium contained in a confined space, the pressure of the liquid increases. This increase in pressure of the liquid is proportional to the applied force. Hence a measure of the increase in pressure of the liquid becomes a measure of the applied force when calibrated.

The main parts of a hydraulic load cell are as follows:

1. A diaphragm
2. A piston with a loading platform (as shown in figure) placed on top of the diaphragm.
3. A liquid medium which is under a pre-loaded pressure is on the other side of the diaphragm.
4. A pressure gauge (bourdon tube type) connected to the liquid medium.

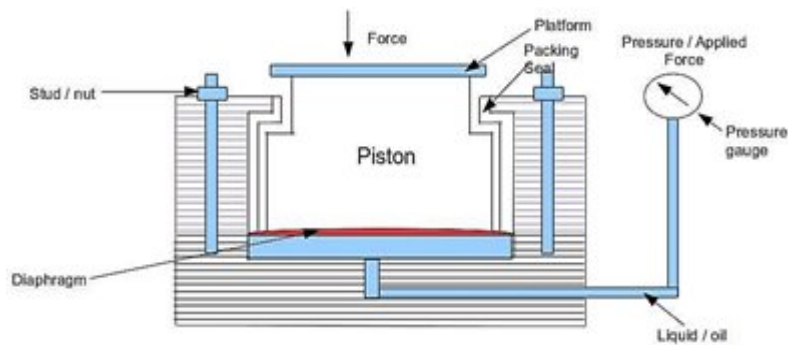


Fig.3.8 Hydraulic Load Cell

Operation of Hydraulic Load Cell

The force to be measured is applied to the piston. The applied force moves the piston downwards and deflects the diaphragm and this deflection of the diaphragm increases the pressure in the liquid medium (oil). This increase in pressure of the liquid medium is proportional to the applied force. The increase in pressure is measured by the pressure gauge which is connected to the liquid medium. The pressure is calibrated in force units and hence the indication in the pressure gauge becomes a measure of the force applied on the piston. Note about Hydraulic Load cell: As the hydraulic load cell is sensitive to pressure changes, the load cell should be adjusted to zero setting before using it to measure force. This hydraulic load cell have an accuracy of the order of 0.1 percent of its scale and can measure loads upto $2.5 \times 10^5 \text{ Kg}$. The resolution is about 0.02 percent.

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3.4.3 PNEUMATIC LOAD CELL

The main parts of a pneumatic load cell are as follows:

A corrugated diaphragm with its top surface attached with arrangements to apply force.
An air supply regulator, nozzle and a pressure gauge arranged as shown in figure.
A flapper is arranged above the nozzle as shown in Figure 3.9.

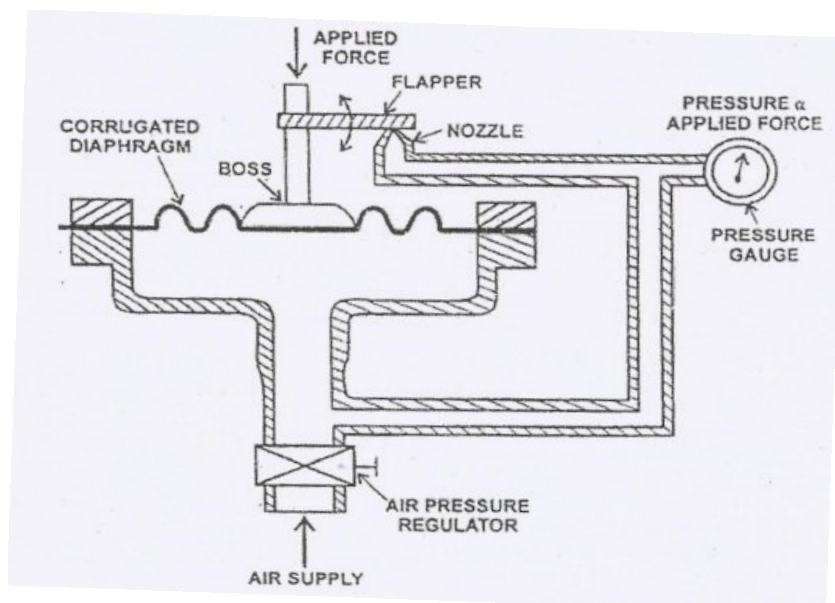


Fig.3.9 Pneumatic Load Cell

Operation of Pneumatic Load cell

The force to be measured is applied to the top side of the diaphragm. Due to this force, the diaphragm deflects and causes the flapper to shut-off the nozzle opening. Now an air supply is provided at the bottom of the diaphragm. As the flapper closes the nozzle opening, a back pressure results below the diaphragm. This back pressure acts on the diaphragm producing an upward force. Air pressure is regulated until the diaphragm returns to the pre-loaded position which is indicated by air which comes out of the nozzle. At this stage, the corresponding pressure indicated by the pressure gauge becomes a measure of the applied force when calibrated.

3.5 TORQUE MEASUREMENT

Torque is the tendency of a force to rotate an object around an axis, fulcrum, or pivot. Just as a force is a push or a pull, a torque can be thought of as a twist to an object.

Torque is the tendency of a force to cause or change the rotational motion of a body. It is a twist or turning force on an object.

Torque is calculated by multiplying force and distance. It is a vector quantity, meaning it has both a direction and a magnitude.

The SI units of torque are newton-meters or N*m.

The symbol for torque is typically τ , the lowercase Greek letter tau. When it is called moment of force, it is commonly denoted by M.

The magnitude of torque depends on three quantities: the force applied, the length of the lever arm connecting the axis to the point of force application, and the angle between the force vector and the lever arm.

$$\tau = r \times F = r F \sin\theta$$

where

τ is the torque vector

r is the position vector (a vector from the origin of the coordinate system defined to the point where the force is applied)

F is the force vector,

$\times$ denotes the cross product,

θ is the angle between the force vector and the lever arm vector.

The SI unit for torque is the newton metre (N·m).

Measurement of Torque is of fundamental importance in all rotating bodies to ensure that the design of the rotating element is adequate to prevent failure under shear stresses. Torque measurement is also necessary part of measuring the power measuring the power transmitted by rotating shafts.

3.5.1 GRAVITY BALANCE METHOD

The method is illustrated in the figure. A mass m is moved along an arm until the value of the torque exerted by the mass balances the unknown torque.

Unknown Torque, $T = Fr$

where, $F = mg$ = force exerted by the mass.

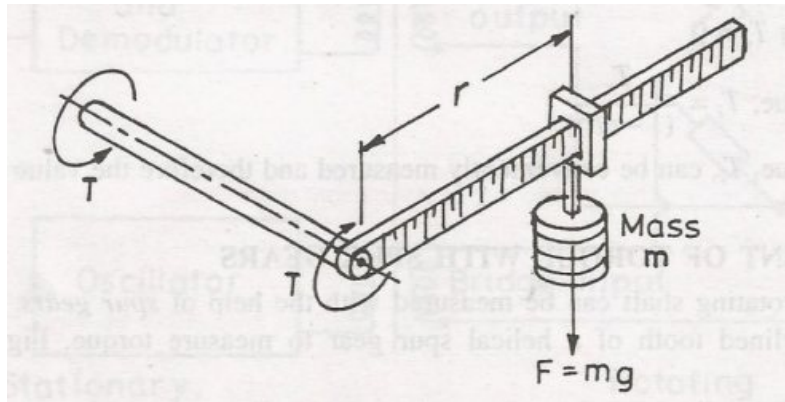


Fig.3.10 Gravity Balance

This method utilizes the movement of a constant mass m , over a variable distance. Alternately, the distance may be kept constant and the unknown torque is balance against variable masses.

In both the cases, the arm must be horizontal, so that the movement arm distance is perpendicular to the line of action of force. The shaft is supported at a bearing and the force acting on the bearing causes a friction torque, leading to error in measurement of torque. This error may be eliminated by arranging to apply equal and opposite force.

3.5.2 OPTICAL TORSION METER

Due to torque, an angular twist (angular displacement) occurs on the shaft between its two ends. This angle of twist is measured by using optical means where in angular deflection of light rays is proportional to twist and hence the torque.

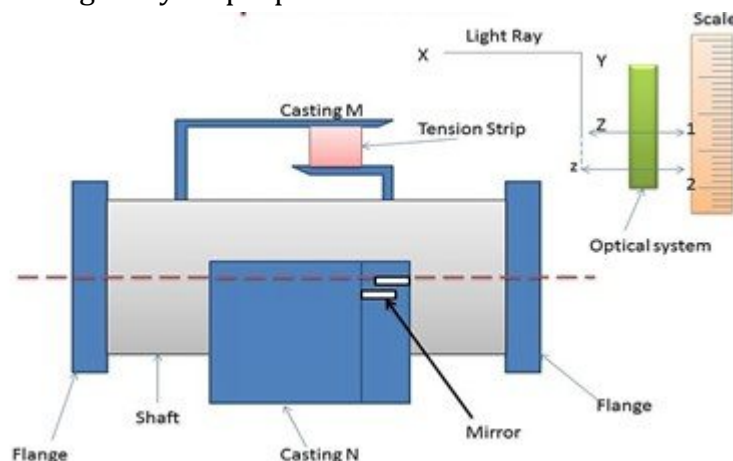


Fig.3.11 Optical Torsion Meter

The main parts of an optical torsion meter as follows:

A shaft is used on which two casting M and N are connected at a known distance.

A tension strip linking the two castings.

Two mirrors which are fitted and aligned on the castings.

A light beam falling on the mirrors, an optical system and a torque calibrated scale.

Operation Of Optical Torsion Meter

When the shaft is transmitting torque, a relative movement occurs between castings M and N, and due to this, the mirrors will change position (partial inclination between the two mirrors) since they are attached to the castings.

As the mirrors are constantly made to reflect a light beam on the torque calibrated scale, due to the changed position of the mirrors, there will be an angular deflection of the light rays which is measured from the calibrated scale.

This angular deflection of the light rays is proportional to the twist on the shaft (relative movement of casting M and N) and hence the torque of the shaft.

Applications of Optical Torsion Meter

It is used in steam turbines and I.C engines

3.5.3 ELECTRICAL TORSION METER

Basic Principle

Due to the applied torque, there is a relative displacement between the two slotted discs. Due to this relative displacement of the slotted discs, a phase shift exists between the pulse generated by the transducers. When these pulses are connected to an electronic unit, it will show a time lapse between the two pulses. This time lapse between the two pulses is proportional to the twist of the shaft and the torque of the shaft.

Description of Electrical Torsion Meter

1. The main parts of an electrical torsion meter are as follows:
2. A shaft connected between a driving engine and a driven load.
3. Two slotted discs attached on either side of the shaft.
4. Transducer (magnetic or photo electric) to count pulses from the slotted disc.

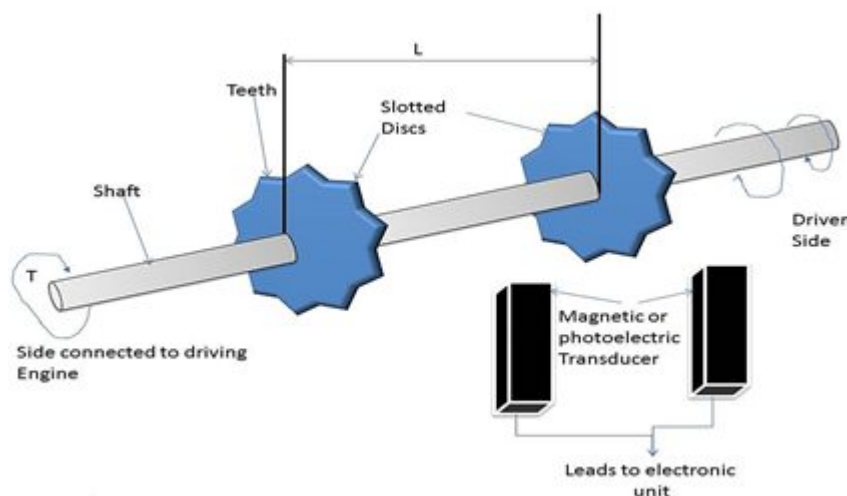


Fig.3.12 Electrical Torsion Meter

Operation of Electrical Torsion Meter

1. The teeth produce voltage pulses in the transducers.
2. When torque is not applied on the shaft, the teeth of the both the discs perfectly align with each other and hence the voltage pulses produced in the transducers will have zero time difference.
3. But when torque is applied on the shaft, there is a relative displacement of the slotted discs due to twist experienced by the shaft and hence the teeth of both the discs will not align with each other and hence the voltage pulses produced in the transducer will have a time difference (that is , time lapse).
4. This time lapse between the pulses of the two discs is proportional to the twist of the shaft and hence the torque of the shaft.
5. A measure of this time lapse becomes a measure of torque when calibrated.

Application of Electrical Torsion Meter

1. Used to measure torque on rotating shafts.

Advantages of Electrical Torsion Meter

1. There are no signal leakage problems.
2. There is no noise creation.

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3.5.4 STRAIN GAUGE TORSION METER

A torque sensor or transducer converts torque into an electrical signal. The most common transducer is a strain gauge that converts torque into a change in electrical resistance. The strain gauge is bonded to a beam or structural member that deforms when a torque or force is applied.

A Strain Gauge is a metallic conductor. When this strain gauge is stretched or compressed, its resistance changes, because of the fact that both length and diameter of the conductor (strain gauge) changes.

The arrangement of the strain gauge torsion meter consists of the following:

- Four bonded wire strain gauges are mounted on a 45° helix with the axis of the shaft rotation. These gauges are placed in pairs diametrically opposite to each other.
- All the four strain gauges are connected to a Wheatstone bridge circuit which is used to measure change in the resistance of the strain gauges.
- The strain gauges are temperature compensated and a change in the resistance of the strain gauges will occur only due to the twist (torsional deflection).

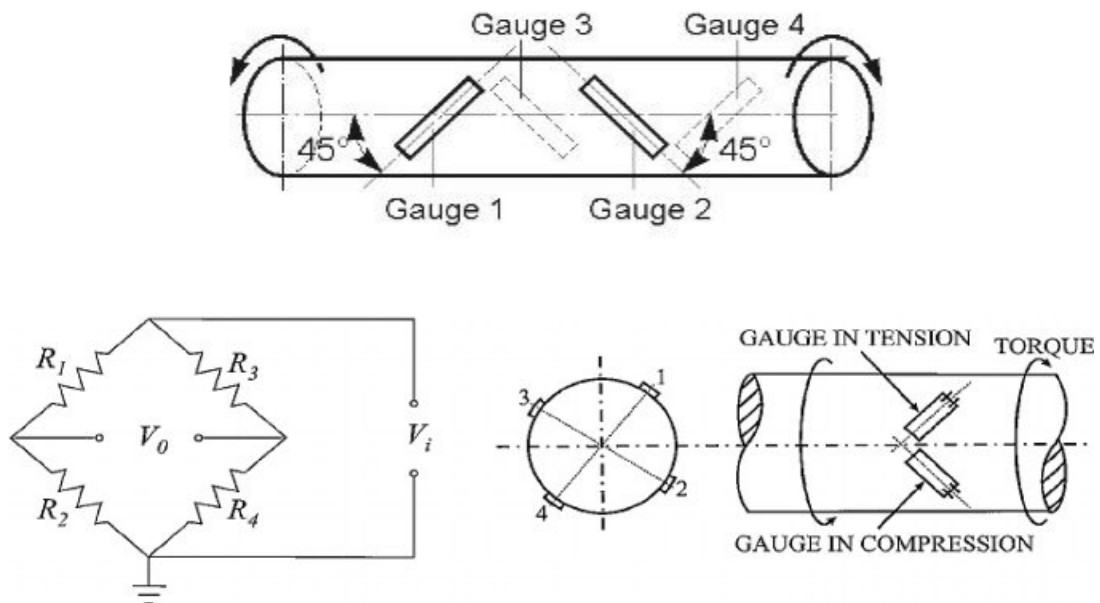


Fig.3.13 Strain Gauge Torsion Meter

Operation

- When the shaft is subjected to a torque, strain gauges 1 and 4 will elongate due to tension and strain gauges 2 and 3 will contract due to compression.
- Due to the tensile and compressive effects on the strain gauge, their resistance will change and this change in resistance of the strain gauges is measured using Wheatstone bridge circuit.
- The change in resistance of the strain gauges will be proportional to the torque on the shaft. Hence a measure of this change in resistance of the strain gauge becomes a measure of torque when calibrated.

Application

- Used to measure torque on rotating shafts.

Advantages

- Fully temperature compensated.
- Sensitivity is high.

Limitations

- It is difficult to connect the bridge circuit to the power source.
- It is difficult to connect the display (galvanometer) to the bridge circuit.

3.6 MEASUREMENT OF SHAFT POWER

The conventional methods of determining the power delivered to (or absorbed by) rotating machines require simultaneous measurement of torque and speed.

A **dynamometer** is a device used for measuring the torque and brake power required to operate a driven machine, usually under test bed conditions.

The dynamometers are classified as follows

1. ABSORPTION DYNAMOMETER

These dynamometers absorb and convert the shaft power to be measured into heat and dissipate to the surroundings.

- (e.g) Prony Brakes
- Rope Brakes
- Hydraulic (or) Fluid Friction Brakes.
- Eddy current Dynamometer.

2. TRANSMISSION DYNAMOMETER

These dynamometers absorb the power to be measured and after measurement convey that power to the surroundings in a useful form (mechanical / electrical).

- (e.g) Torsion and Belt dynamometers.
- Epicyclic train dynamometers.
- Strain gauge dynamometers.

3. DRIVING DYNAMOMETER

These instruments measure power and also supply energy to operate the tested devices.

- (e.g) Electric Cradled Dynamometers.

MECHANICAL BRAKES

Prony Brake and Rope brake are the two widely used mechanical brakes.

3.6.1 PRONY BRAKE DYNAMOMETER

Prony Brake is one of the simplest dynamometer for measuring power output (brake power). It is to attempt to stop the engine by means of a brake on the flywheel and measure the weight which an arm attached to the brake will support, as it tries to rotate with the flywheel.

It consists of two wooden blocks placed around a pulley. The engine shaft whose shaft power to be measured is connected with this pulley. The wooden blocks are clamped by means of bolts and nuts. A helical spring is used to adjust the pressure on the pulley to control the speed. The upper block has a long lever and carries a weight (w) at its outer end. A counter weight is connected on the other side of the lever. Stops are used to limit motion of the lever.

While in operation, the long end of lever is loaded with suitable weight. The nuts are tightened until the engine shaft runs at a constant speed. The lever is maintained in

F = Frictional resistance between the pulley and the blocks

N – Engine speed, r.p.m

D – Diameter of the brake wheel, m

d – Diameter of the rope, m

And (D+d) – effective diameter of the brake wheel.

$$\begin{aligned}\text{Brake Power B.P} &= (W - S) \pi (D+d)N / 60 \times 1000 \text{ KW} \\ &= (W - S) \pi DN / 60 \times 1000 \text{ KW} \\ &= [T \times 2\pi N / 60 \times 1000] \text{ KW}\end{aligned}$$

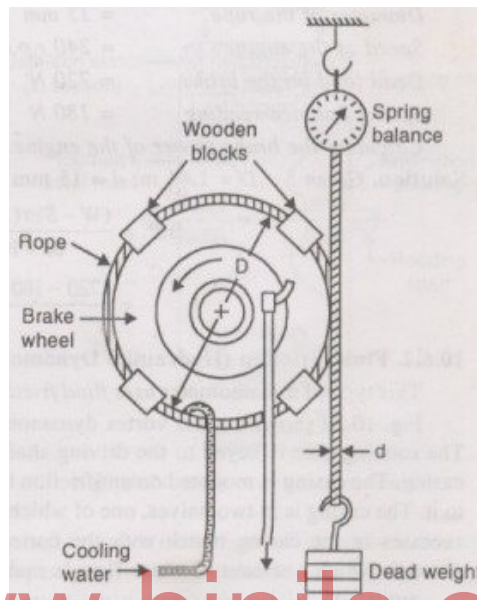


Fig.3.15 ROPE BRAKE DYNAMOMETER

- It is necessary to keep the brake wheel cool with soapy water.
- It is cheap, easy to construct and not accurate.

3.6.3 EDDY CURRENT DYNAMOMETER

The Eddy Current Brake utilizes the power loss produced on account of eddy currents and therefore acts as an absorption type dynamometer.

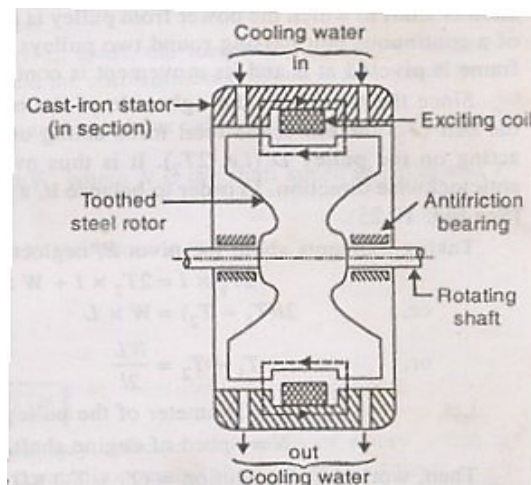


Fig.3.16 Eddy Current Dynamometer

Construction

It consists of a toothed steel rotor. The rotor is mounted on the shaft of a test engine. The rotor rotates inside a stator. The clearance between the stator and rotor is very small. The stator carries an exciting coil, which is energized by an external DC source. The stator is cradled on antifriction bearings. The stator is also provided with a brake arm to which a spring balance is connected.

Working

When the dynamometer is operated, the rotor turns. Eddy currents are induced in the rotor this eddy currents oppose the rotation of the rotor. The moment of resistance is measured by the brake arm. From the moment we can calculate the torque and shaft power. The mechanical power supplied to the dynamometer shaft is converted to heat which is carried away partly by air and partly by water circulation. Usual limit of the dynamometer is 300 H.P., and the speed limit is 6000 r.p.m.

Advantages

- i. Good control at low rotating speeds.
- ii. Comparatively small size for a given capacity.
- iii. Suitable for a higher speed range.

3.6.4 FLUID FRICTION DYNAMOMETER

Construction

This type of dynamometer uses fluid friction for dissipating the input energy. It consists of a rotating disc and a stationary casing. The rotating disc is connected with the driving shaft of the engine. Thus the disc revolves inside the stationary casing. The casing is mounted on anti-friction bearings and has a brake arm & a balance system attached to it. The casing is in two halves, each one is placed on either side of the rotating disc. Semi elliptical recesses in the casing match with grooves in the rotating disc. This forms chambers through which water can be supplied.

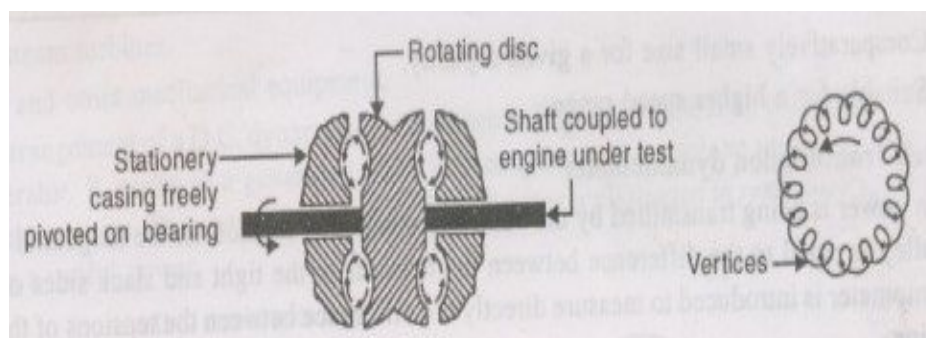


Fig.3.17 FLUID FRICTION DYNAMOMETER

Working

While the brake is operating, the water flows in a helical path in the chamber. This sets up vortices and eddy currents in water. This turns the dynamometer casing in the direction of rotation of the shaft. This action is opposed by the brake arm and

balance system. Thus the torque is measured. The control of braking action is carried out by varying either the quantity of water or its pressure, or the distance between the rotating disc and the stationary casing. The power absorption (P) in this dynamometer is

$$P \propto N^3 (d)^{1/5},$$

Where N is the rotational speed and d is the rotor diameter.

Usual power limit of this dynamometer is 25000 H. P. and the speed limit is 10000 r.p.m.

ADVANTAGES

- In addition to braking action, the supply of water provides cooling effect.
- High absorption capacity at low cost and in a small space.
- Hunting effects can be avoided by providing dashpot-damper system.

3.6.5 DC DYNAMOMETER

The most versatile and accurate dynamometer is the DC Dynamometer. This is a cradled dynamometer and is widely used for power and torque measurements of

- Internal combustion engines
- Small steam turbines
- Pumps and other mechanical equipment.

The machine acts as both motor and generator. When the DC Machine is coupled to the machine under test which is a power generating machine, it is made to work as a generator. The generated power is of the DC Generator is dissipated in the resistance grids or may be recovered as useful power. Thus the DC machine can act both as absorption dynamometer and a transmission dynamometer.

The force and torque is measured through a force / torque arm attached to the casing of the DC machine. The output voltage and hence the power can be controlled by controlling the field excitation of the generator.

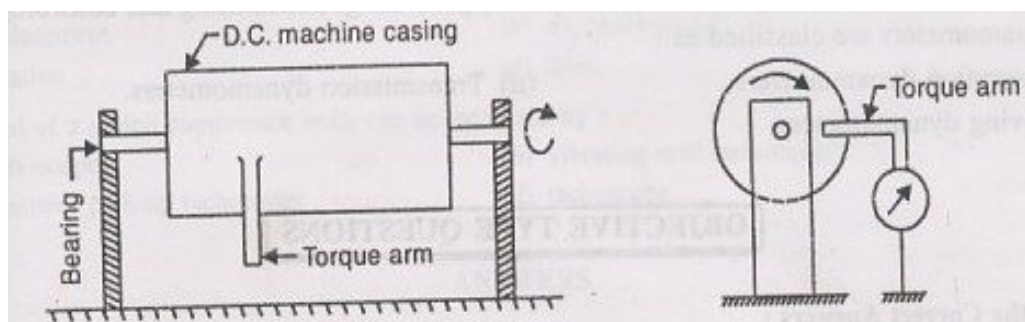


Fig.3.18 DC Dynamometer

When the DC machine is made to work as a DC motor, it may act as a driving dynamometer. Thus the DC machine may drive the test machine which is a power absorbing device like a pump.

The output power of the pump may be smoothly controlled by controlling the speed of the DC motor by varying either the armature voltage or field current.

The control panel of the dynamometer is provided with ammeter and voltmeter, so that the output power can be computed.

When used as an absorption dynamometer, the range of DC Dynamometer is 5000 h.p.

REVIEW QUESTIONS

Part A (2 marks)

1. Define force and mention its units.
2. What is Torque and mention its units?
3. What is a Dynamometer?
4. What is a Torsion meter?
5. List the different methods of Torque measurement.
6. What are the different types of Dynamometer?

Part B (3 marks)

1. What are the advantages of using Unequal Arm Balance?
2. How elastic elements are used in Force measurements?
3. Write short notes on Hydraulic Load Cell.
4. Brief on gravity balance method of torque measurement.
5. Write short notes on Rope Brake Dynamometer.

Part C (10 marks)

1. Explain the construction and operation of Pendulum Scale with a neat diagram.
2. With a neat diagram explain the construction and working of Strain Gauge Load Cell.
3. Explain the Electrical Torsion meter with a neat diagram.
4. Explain the Optical Torsion meter with a diagram.
5. With necessary diagram explain the Fluid Friction Dynamometer.
6. Explain the DC Dynamometer with a neat diagram.
7. Explain the Eddy Current Dynamometer with a neat diagram.

UNIT IV - MEASUREMENT OF pH & GAS ANALYSIS

pH Definitions – Electrodes: Hydrogen electrode - Calomel electrode - Quinhydrone electrode - Antimony electrode - Glass electrode

Gas Analyzer (principles of operation and working): Oxygen Analyzer – paramagnetic oxygen analyzer – CO analyzer - SO₂ analyzer.

4.1. DEFINITION OF pH:

The number of gram ions of hydrogen present in one litre of the solution is called the hydrogen ion concentration of the solution. The acidity or basicity of a solution can be expressed in terms of hydrogen ion concentration. PH is defined as the negative logarithm of the hydrogen ion concentration. It is expressed as $\text{pH} = -\log_{10} (\text{H}^+)$

pH = 7 solution is neutral

pH > 7 solution is basic

pH < 7 solution is acidic

4.2. ELECTRODE:

Electrode is a material or a metallic rod or bar through which electrons flow. There are two types of electrodes. They are anode and cathode.

4.2.1. ANODE:

Anode is the electrode at which oxidation occurs. So this electrode is called as oxidizing electrode.

4.2.2. CATHODE:

Cathode is the electrode at which reduction occurs. So this type is called as reducing electrode.

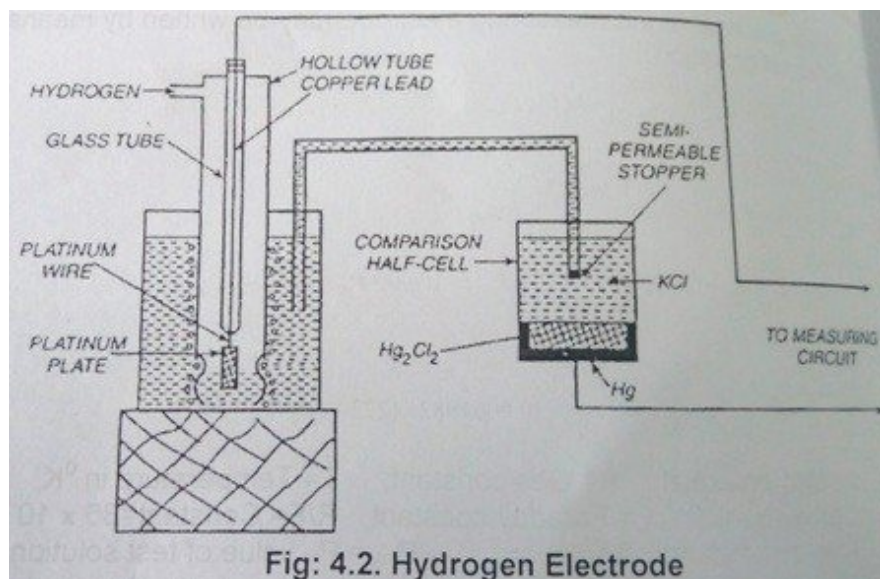
4.2.3. TYPES OF ELECTRODE:

- Reference Electrode
- Indicator Electrode

4.3. PRINCIPLE OF OPERATION AND CONSTRUCTION OF HYDROGEN ELECTRODE:

The Hydrogen Electrode consists of a hollow tube which is opened at the bottom and is immersed in the solution whose pH value is to be measured. The platinum plate is covered with platinum black is immersed into this hollow tube and is joined to the platinum wire which is welded to the glass tube. This platinum wire is joined to a copper lead. The hydrogen gas is fed to the outer tube and passes through the lower aperture placed at level, half way up the platinum plate, the hydrogen electrode is connected to the comparison half cell by an electrolytic solution. The cell is filled with an inert salt KCL and closed by a semi permeable stopper. This type of connection

lowers the diffusion potential up to 1-2mv which arises at the boundary of the two solutions and introduces an error into the result of the measurement.



The standard hydrogen cell with the normal concentration of Hydrogen ions can be employed as the comparison cell. The potential of the standard Hydrogen electrode is tentatively taken as zero, in relation which the potential of the electrodes are determined.

$$E = E^0 - 0.0001982 \times (273 + t) \times (\text{PHS} - \text{PHM}); \text{PHS} = 0; E^0 = 0$$

$$E = E^0 - 0.0001982 \times (273 + t) \times (-\text{PHM}) = 0.0001982 \times (273 + t) \times (\text{PHM})$$

$$\text{PHM} = E / 0.0001982 \times (273 + t)$$

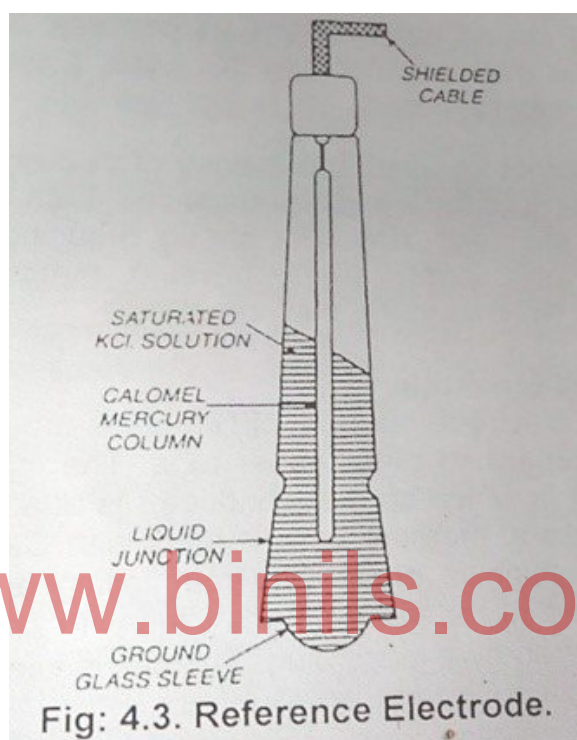
This method is mainly used in laboratories or pH measurements from 1 to 14. Its use is limited to industrial to industrial applications, because of some of the operational defects, which are

1. Necessity of saturating with hydrogen.
2. Poisoning of electrode in consequence by the platinum black or some component parts of the solution.

4.4.1. Measuring and Reference electrode:

The electric potential developed on a Hydrogen electrode in a solution is caused by the concentration of Hydrogen ions in the solution. By measuring this electric potential developed on Hydrogen electrode, pH of the solution can be measured. There is no method by which the absolute value of the potential developed on hydrogen electrode could be determined. It is always the potential difference between the two objects or between the two points on the same object. Hence it is possible only to measure the relative potential of hydrogen electrode as compared to the potential of hydrogen electrode as compared to the potential of some reference electrode whose potential is known.

The reference electrode is designed to produce a constant potential regardless of the solution in which it is immersed. This constant potential is used as a reference point from which the variable potential produced by other electrode is measured. An ideal reference electrode is one which when placed along with the hydrogen electrode in the test solution, gives a fixed potential which is independent of the composition of the solution. But in practice, it is not possible, but the potential developed in a reference electrode (which uses a metal as a reference) due to the disturbing effect caused between and the solution should be minimum.



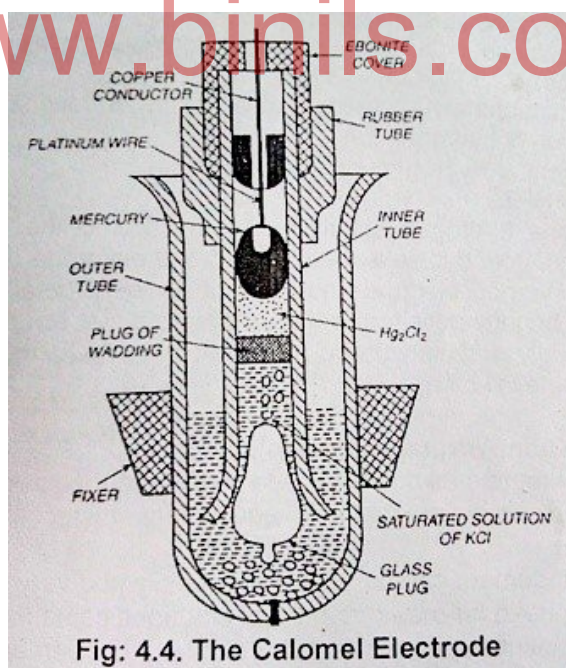
A reference electrode usually consists of mercury and calomel (mercurous chloride- Hg_2Cl_2) immersed in the saturation KCL as a connecting medium, the junction potential which is developed due to the connection between different metals or a metal and a liquid or between two liquids will be very little because of minimum disturbing effect on the potential. The conducting path between the electrode and the process stream can be maintained by allowing a very small amount of electrolyte to flow out of the reference by means of the porous plug. Normally PH of the solution to be measured is kept in an open tank. In these case, the electrolyte flows into the solution by the test solution by the pressure developed due to Hydraulic head of the electrolyte. But sometimes PH of the test solution under pressure is also to be measured. In this case, external air pressure is applied to the reference electrode. This pressure difference makes the electrolyte to flow into the process stream. The process stream pressure should not be variable and must be regulated by pressure regulator. When the Reference electrode is fully submerged in the test solution, the flow of electrolyte can be done with pressure equalizing tube.

4.4.2. Limitations:

1. It requires a hydrogen generation station.
2. High care should be taken to avoid poisoning of platinum black. This lead to constant replacement of platinum black causing maintenance and efficiency problems.
3. H_2 gas should be saturated with vapors of the test solution to avoid alteration of concentration of test solution.
4. H_2 gas to be purified from all impurities including O_2 .
5. Rubber tubing should not be used as it contains Sulphur which will contaminate the platinum black. In this case, Polythene or polyvinyl chloride (PVC) tubing is used.

4.5. CALOMEL ELECTRODE:

The calomel electrode consists of two glass tubes with one placed inside the other, separated by a rubber tube. The inner tube is closed with ebonite cover, in which copper conductor is attached to a platinum wire which is dipped in mercury is in contact with calomel (mercurous chloride - $HgCl_2$), Which is prevented from failing down by a plug of wadding. The bottom of the inner tube is filled with saturated solution of KCl and this is covered by a glass plug .the cell is fixed in the pick up by using tapered cork type fixer made of rubber.



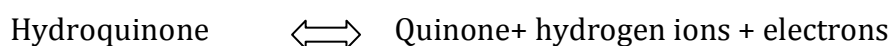
The electrode solution forms a conductive salt bridge between the element and sample solution in which mercury and reference electrode is emplaced. For a stable electrical connection between internal metallic element and sample junction, a small but constant flow of electrolyte solution is maintained through a liquid junction in the tip of the outer body of the electrode.

These electrodes are used in laboratories for pH measurements. However its useful life is known to be short at temperature above 70 degree Celsius. The sensors may contain Resistance Thermometer for the temperature compensation, which controls temperature co-efficient of the electrode automatically. Although this electrode is most commonly used electrode, sometimes it is necessary to use other type of Reference electrodes.

1. Ag/AgCl electrode is recommended as a Reference electrode at high temperature
2. Hg/mercurous sulphate is recommended with salt bridge solution of K_2SO_4 is used in test solution that must not contain chloride ions.
3. Hg/Hg₂Cl₂ with a salt bridge solution of KNO₃ is used in test solution, which does not contain chloride ions.
4. Hg/Hg₂Cl₂ with a salt bridge solution of Lithium chloride is used for the measurement of pH in non-aqueous solutions.

4.6. QUINHYDRONE ELECTRODE:

One molecule of hydroquinone and one molecule of quinone make a molecule of Quinhydrone. So Quinhydrone is considered to consist of two chemicals, hydroquinone and quinone changes to quinone and this change is reversible as shown below.



The equations are similar to the equations representing the dissociation of hydrogen ion.



This as a hydrogen electrode is sensitive to the hydrogen ion concentrations around it, so is the Quinhydrone electrode.

A Quinhydrone electrode consists of an inert metal such as gold and platinum brightly polished. It is dipped into a test solution into which a certain quantity of Quinhydrone has been added. Quinhydrone is very sparingly soluble in water.

4.6.1. Advantages:

1. Platinum metal need not be coated with platinum black etc.
2. Electrode potential reaches equilibrium faster than that in hydrogen electrode
3. The arrangement can be set up very easily.
4. It can be used for measuring pH of solutions containing substances such as nitric acid, nitrates, copper and lead salts, unsaturated organic acids and alkaloid drugs.

4.6.2. Disadvantages:

1. Quinhydrone electrode is susceptible to salt error if the concentration of the electrolyte in test solution is greater than $M/10$. However, this defect can be rectified by saturating the solution with either Hydroquinone or Quinine and quinhydrone.
2. Quinhydrone electrode can be only be used up to pH 8.5, that is in acidic or near neutral solution. It is because quinhydrone is itself weak acid and when added to alkaline solution it dissociates into ions, upsetting the pH of the test solution.

4.7. ANTIMONY ELECTRODE:

When antimony is put in water, it gets slightly corroded and a very slightly soluble oxide film is formed on its surface. As this oxide is very slightly soluble in water so water is very soon saturated with antimony going into solution in water. This antimony metal rod in presence of antimony ions dissolved in water develops an electrode potential. Due to hydrolysis effect, the concentration of antimony ions in water depends upon the hydrogen ion concentration in water. Thus electrode potential depends upon the hydrogen ion concentration that is pH antimony electrode consists of only an antimony rod to which an electrical lead is attached.

4.7.1. Limitations Antimony electrode:

1. Antimony electrode takes time to develop its potential.
2. The potential developed on antimony electrode is affected by the presence of other substances in test solution.
3. The surface of the electrode has to be continuously scraped to keep it clean in order to get best results.

4.8. GLASS ELECTRODE:

Its action is based on the principle, when a thin membrane of glass is interposed between two solutions, a potential difference is observed across the glass membrane, which depends on the presence of ions in the solutions. Depending on the composition of glass the response may be to H ions or it may be to cations. The selective response of certain glass compositions to H ions has led to development of pH responsive glass electrode.

It consists of a thin walled bulb made out of a special glass and to it is sealed with a stem of soda or lead glass. The bulb is generally blown from a glass of composition 72% silica, 22% of Na_2O and 6% CaO . This type glass has low melting point and high electrical conductivity but still the electrical resistance is usually from 50-200 Mega ohms. Glass bulb is filled with $N/10$ HCl and a silver wire coated with silver chloride dipped into it. The conditions inside the bulb is standardized as the potential developed across the glass film is proportional to the H ions concentration, i.e. pH of both the liquids which are in contact with two glass surfaces on either side of the glass.

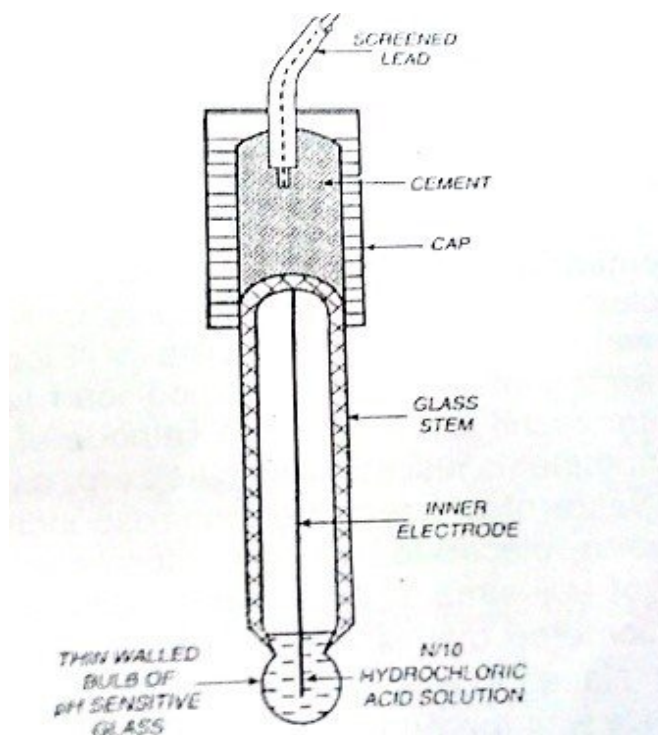


Fig: 4.5 Glass Electrode

When similar solution is placed on either side of glass electrode, there will be a potential of $\pm 2\text{mV}$ or even lower is observed. This is called Asymmetric potential, which is due to difference in strain on inner and outer surface of the bulb. Thus the potential of Glass electrode depends on the nature of inner electrode, composition of inner solution and Asymmetric potential.

4.8.1. Limitation of Glass electrode:

1. Glass electrode gives accurate result in 1-9 pH range. Below pH1, Acid error and above pH 9, Alkali errors are introduced. Errors will be large above pH 11 and below pH 1 and it depends on temperature, type of glass and the composition of the solution.
2. Highly acidic, alcoholic, high temperature and the presence of colloidal particles introduce errors, when this type of electrode is used.
3. A glass membrane exhibits high electrical resistance. The measurement circuit therefore should have high input impedance.
4. Due to large change in electrical resistance with temperature, it is useful when temperature is within 60 degree Celsius.

4.9. GAS ANALYSERS

Gases are distinguishable by their

- i) Thermal properties
- ii) Magnetic susceptibilities.
- iii) Absorption / Emission of electromagnetic radiation.
- iv) Chemical or Electrochemical reactions.

4.10. Oxygen Analysis

Analysis of the presence of oxygen is important for two types of applications

- i) Applications where oxygen is necessary for oxidation and combustion.
- ii) Applications where the contamination of oxygen needs to be prevented (such as in the production of pure inert gases).

The oxygen specific analysers can be divided into three categories namely,

- i) Paramagnetic analysers.
- ii) Electrochemical analysers
- iii) Catalytic combustion analysers.

4.11. Paramagnetic Oxygen Analyser

A paramagnetic substance, is drawn to the stronger area of magnetic field while a diamagnetic substance behaves just in the opposite way. Among the gases oxygen(O_2), nitrogen monoxide(NO) and nitrogen dioxide(NO_2) are paramagnetic, hydrogen and carbon monoxide are very low paramagnetic, while the other gases are diamagnetic at ordinary temperature as shown below.

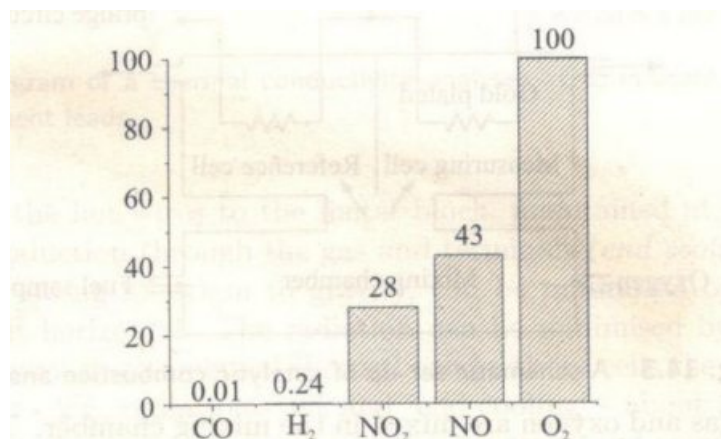


Fig : 4.6. Relative Susceptibilities of Paramagnetic Gases

Types of paramagnetic oxygen analysers are

- i) Deflection / Magnetic force type
- ii) Magnetic wind type

4.11.1. DEFLECTION TYPE / MAGNETIC FORCE TYPE OXYGEN ANALYSER

It consists of a glass dumbbell suspended between the poles of a permanent magnet by a quartz fiber. The dumbbell is filled with nitrogen gas (or some other gas of low magnetic susceptibility). The magnetic pole pieces are in wedge shape, so that the field between them is non-uniform.

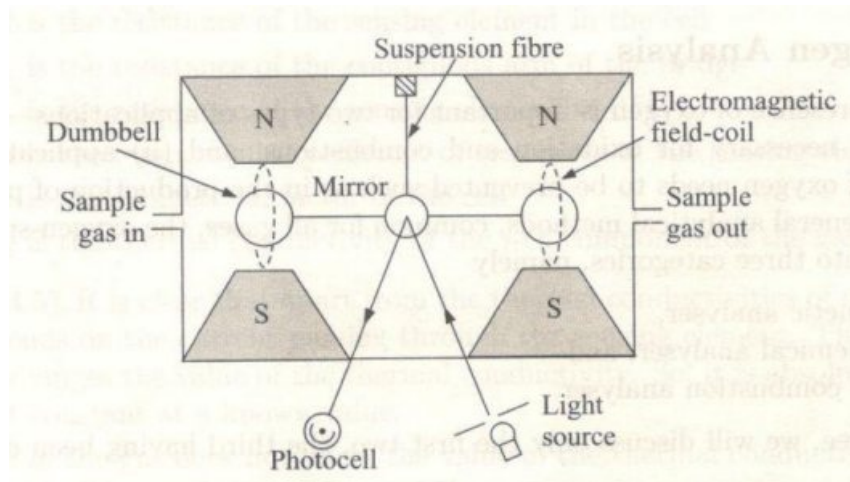


Fig : 4.7. Deflection Type Paramagnetic Oxygen Analyser

Without the sample gas, the dumbbell stays slightly away from the strongest part of the magnetic field. As the sample gas is let in, its oxygen content occupies the strongest part of the magnetic field. Thus the dumbbell is displaced further. This causes the deflection of the light incident on the mirror in the suspension fiber.

Displacement of the dumbbell upsets the light balance in the photocell. This unbalance produces a proportional current in the electromagnetic field coils. This current brings back the dumbbell to its original position. This proportional current can be calibrated in terms of the percentage of oxygen content in the sample gas.

4.11.2. LIMITATIONS

- i) This instrument has to be installed on a vibration free pedestal
- ii) Sample gas should be free from dirt.

4.11.3. MAGNETIC WIND TYPE OXYGEN ANALYSER

According to Curie's law, the paramagnetic susceptibility X_p , of a substance depends on the absolute temperature T .

$$\text{i.e., } X_p = C / T \quad \text{where 'c' is a constant.}$$

Thus the paramagnetic property decreases with increasing temperature. This property of paramagnetic oxygen is used to generate a flow which has been termed as magnetic wind.

The following figure shows the schematic diagram of such magnetic wind generating oxygen analyser.

The instrument consists of a glass ring. In the middle of the ring a glass tube is present. This horizontal glass tube houses two wound resistors. These wound resistors form the two arms of a bridge. One of the wound resistors is placed between the pole pieces of a magnet. A current is passed through the resistor, so that they produce heat.

In the absence of oxygen in the flue gas flowing through the ring, the wound resistors are heated to the same extent. The Wheatstone bridge to which the resistors are connected remains balanced.

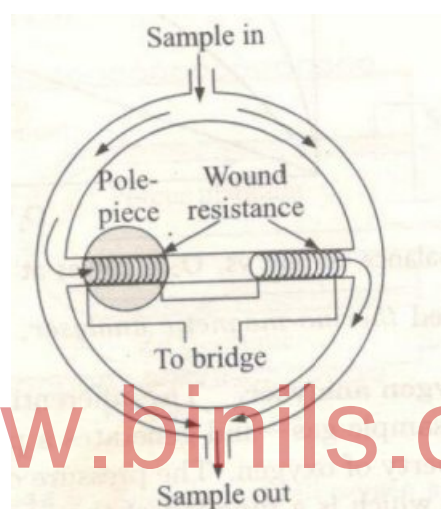


Fig : 4.8. Magnetic Wind Type Paramagnetic Oxygen Analyser

If the sample gas contains oxygen, it is attracted (drawn) to the left resistor, where it gets heated and lose some susceptibility. Now the nearest cold gas on the left having more susceptibility will push the hot gas towards right side and occupy the magnetic field. Thus a magnetic wind is generated from the left to right of the horizontal tube.

As a result, the left resistor region becomes colder than the right one, thus its resistance value decreases. This makes the bridge out of balance. The resulting bridge unbalance current will be proportional to the percentage of oxygen in the sample gas.

4.12. CO ANALYSER

Thermal conductivity gas analyser is used to measure the amount of CO in the flue gases.

Here the flue gas is first passed through one thermal conductivity chamber and then into an apparatus that converts CO into CO₂. The resultant gas is then passed through a second thermal conductivity chamber. Both chambers consists resistors. These resistors are connected with two arms of a Wheatstone bridge circuit.

In the absence of flue gas the resistance value of both resistors will be same and the bridge is balanced. When the flue gas is passed through the chambers the resistance values of both the resistors differ because of the different thermal conductivities of the gases (CO and CO_2). The difference in the output of the two chambers is due to the carbon monoxide (CO) present in the flue gas.

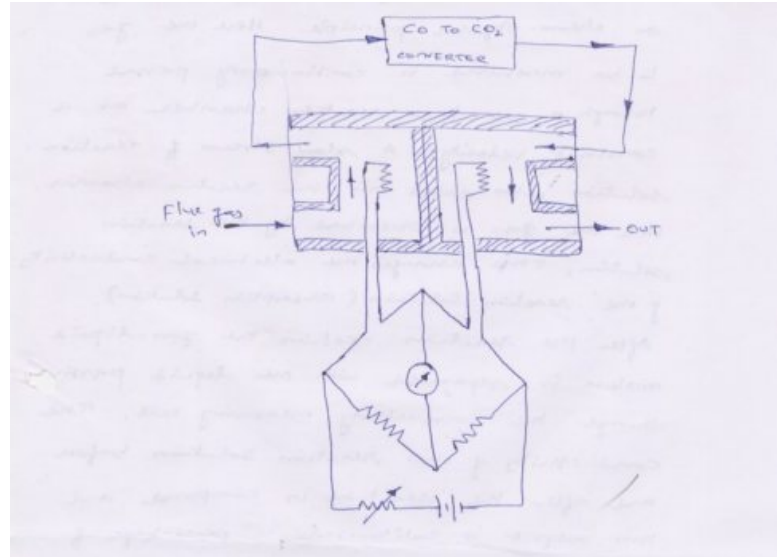


Fig : 4.9. CO Analyser

4.13. SO_2 ANALYSER

The SO_2 Analyser shown here works on Chemo-Physical principle. The gas to be measured is continuously passed through a jet to a reaction chamber at a constant velocity. A slow stream of reaction solution also enters into the reaction chamber. Here the gas is absorbed by the reaction solution. This changes the electrical conductivity of the reaction solution (absorption solution).

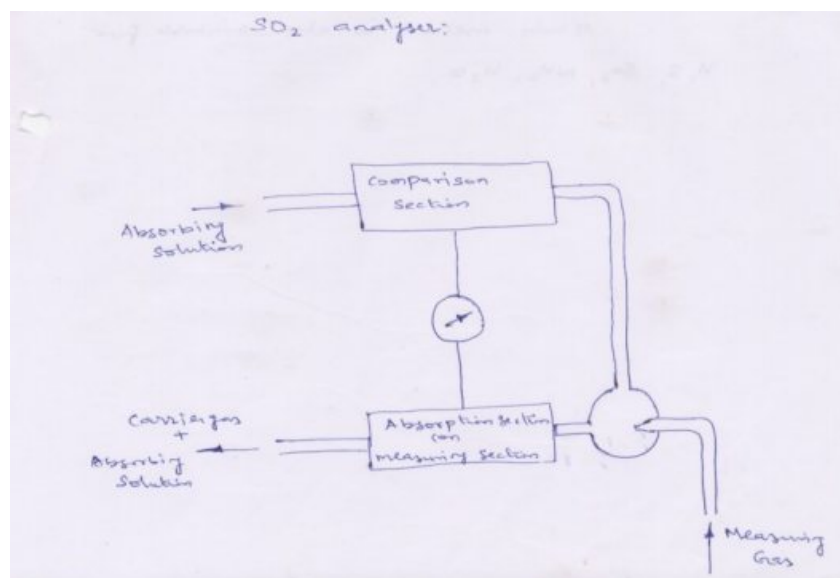


Fig : 4.10. SO_2 Analyser

After the reaction section the gas liquid mixture is separated with the liquid passing through the conductivity measuring cell. The conductivity of this reaction solution before and after the reaction is compared and this output is calibrated in percentage of SO₂ gas.

This method is also suitable for H₂S, SO₂, and NH₂ analysis.

Review Questions: UNIT – IV

PART-A

1. Define pH?
2. What is Anode?
3. What is Cathode?
4. Define types of electrode?
5. What is electrode?
6. What is a Gas Analyser?
7. What is the expansion of SO₂?
8. What is CO Analyser?

PART-B

1. What is the disadvantage of hydrogen electrode?
2. What is Quinhydrone electrode?
3. What are the advantages of Quinhydrone electrode?
4. Draw the diagram of calomel electrode?
5. Briefly explain SO₂ analyser.

PART-C

1. With a neat diagram explain the operation of Hydrogen electrode.
2. Draw and explain the operation of calomel electrode.
3. With a neat diagram explain the operation of glass electrode.
4. With a neat sketch explain the operation of Quinhydrone electrode.
5. With a neat sketch explain the operation of Antimony electrode.
6. With a neat diagram explain the working of Oxygen Analyser.
7. With a neat diagram explain the principle of operation and working of Paramagnetic Oxygen Analyser?
8. Explain the SO₂Analyser in detail.

UNIT V - CHROMATOGRAPHY AND SPECTRAL METHOD OF ANALYSIS

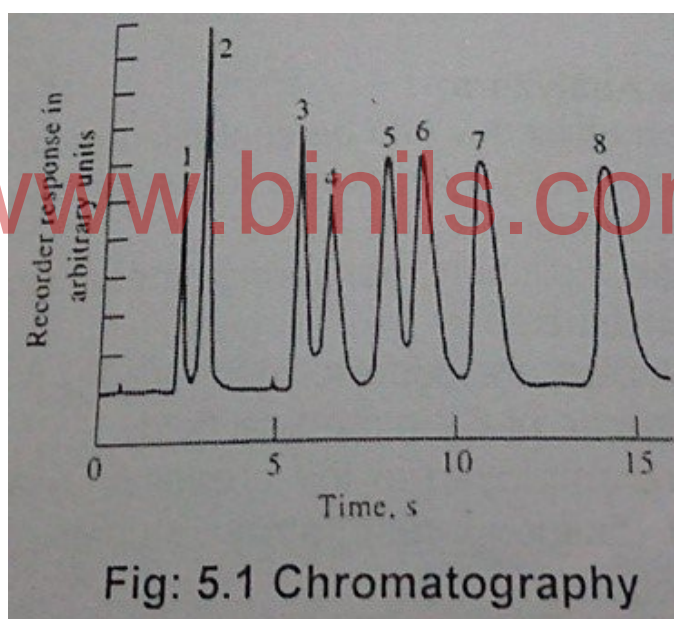
Chromatography: Definition – Classification - Principle of operation and construction - Gas Chromatography - Liquid Chromatography - Retention time -Dead time – Chromatogram - significance and advantages of chromatography

Detectors: Principle of operation and construction of TCD, FID, FPD, ECD

Spectral Analysis: EMR Spectrum- Beer's law- IR/UV spectrophotometry - General description- range of IR/UV radiation - measurement of IR/UV radiation - Instrumentation- IR/UV radiation sources- monochromator- Sample handling.

5.1. DEFINITION:

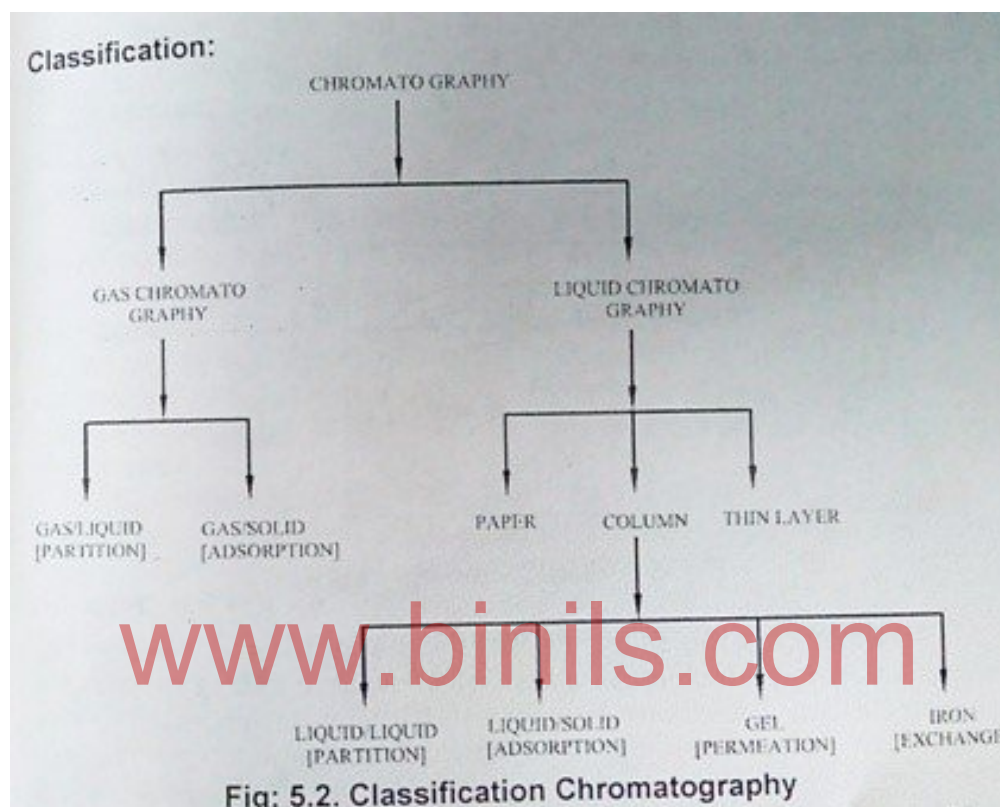
Chromatography is a physical method of separation of the components of a mixture by distorting between two phases, of which one is a stationary bed large area and other a mobile phase that percolates through or along the stationary phase.



The process of chromatography separation involves transports of a sample of the mixture through a column. For this purpose, the mixture may be a solute adsorbed or liquid and it transports the constituents of the mixture through the column. During such transport the materials in the stationary phase, i.e. column exercise selective retardation on the various components of the sample. The retraction may to be adsorption, soluatibility, chemical bonding and polarity of molecular filtration of the sample. Therefore, the components of the mixture tend to move through at different effective's rates and thereby result in tending to segregate into separate zones or bands. The least retarded components emerge first, and the most retained component elutes last. Partition between the phases exploits difference in the physical and chemical properties of the components in the sample.

In the general all chromatography process isolates, detects and characterize these bands at same usually at column exit. Upon emerging from the column, at this place, individual components registers a series of signals which appears as successive peaks as above a basic line on the recorded curve called chromatography. The area under the peak gives a quantitative indication of the particular component.

5.2 Classification :



Based on the mobile phase, chromatography can be classified into

1. Gas Chromatography
2. Liquid Chromatography

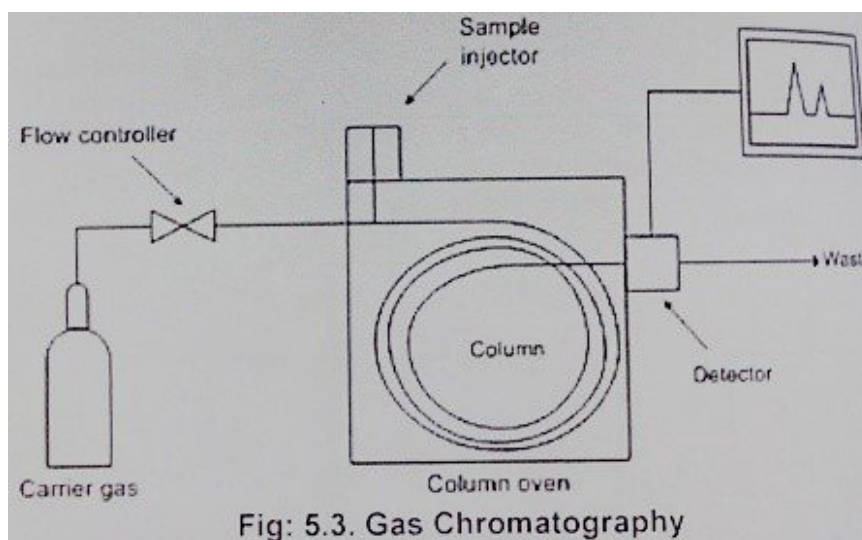
5.3. Gas Chromatography:

The basic parts of Gas chromatography are,

1. Carrier gas along with pressure regulator and flow meter.
2. Sample Injection system
3. Chromatography column thermal compartment
4. Detector system
5. Strip-chart recorder

The carrier gas, normally nitrogen, argon or helium is usually available in a compressed form in a cylinder fitted with a suitable pressure regulator. The gas is conducted from the cylinder through a pressure regulator, to a sample injection system maintained at a certain temperature, which is such that it ensures rapid vaporization but not thermal degradation of the solute. Gas and Liquid samples are almost always injected by a syringe through a self-sealing silicon rubber diaphragm in the injection

port. The solute vapor mixes almost instantaneously with the flowing carrier gas and is swept into a chromatographic column, which is the heart of chromatography.



It is there that the different solutes in the vapourised sample are separated from each other by virtue of this different interaction with the column packing. The column is maintained at the another temperature. This temperature determines the time for the passage of the solutes emerging individually and enters the detectors, which produces an output signal is fed to the recorder and a plot of time-signal amplitude called chromatogram is obtained. This record is used to determine the identity of the components in mixture and their respective concentration. The various parts of a Gas chromatographic system are described below.

Carrier Gas supply system comprises a needle valve, flow meter and a pressure gauge. The carrier gas affects the column and detector performance. The choice of carrier gas is determined by the type of detector, separation column and easy availability. The pressure of the contaminants in the carrier gas affects the column performance and detector performance. The rate of gas affects the detector and hence regulation is necessary.

Sample injection system purpose is to introduce a quantity of sample to be analyzed into the carrier gas stream. The transfer of sample should be made rapidly to ensure that hydrogen sample occupies smallest column volume and layer prevent excessive peak broadening, which affects over all resolution of the system. Sample can be introduced in gaseous, liquid or solid status. The purpose of sample injection system is to insert, volatilize as more resulting gaseous surfaces into the column.

Liquid sample is to injected liquid samples with a micro-syringe through a silicon rubber septum; syringes of variable capacities are available but generally employed for injection of samples between 0.1-10 μ l. sample is injected into the hot zone of the column, so that the liquid gets rapidly transferred to the gashouses state.

Gas sample are injected by a gas tight syringe suitable for delivering 0.1-10ml of sample. They are difficult to handle and often cause inaccuracies. Reduction in sample volume is necessary when working with capillary column. This is accomplished by an injected splitter otherwise called as stream splitter, where typically 1 μ l. sample is injected but only 0.01 μ l enters the capillary, remainder is vented. This technique prevents column overload, but wastes a significant portion of the sample.

Chromatography column is the heart of a gas chromatography, where the fundamental process of separation takes place. As the sample moves through the column, separation takes place and band maintains its shape, which is detected by the detector and recorded as chromatography peak. The degree of peak broadening with respect to time and columns are commonly used.

Packed column is a packed with suitable material, which perform the separation. Columns may be made from any tubular tubings of glass, stainless steel or copper. For moderate temperatures, PVC tubes are used. Internal diameter of the column may be 1-50m which creates problem in packing. Therefore very large columns are best constructed by coupling short sections of tube length less than 3m and of inner diameter of 1.6 - 9.5mm.

Capillary columns are open tubular column of having approximately 0.25mm in diameter and their length may be 30-300mm. Very high efficiency are achieved with this column, since the cross diffusion of sample molecules is minimized by the narrow diameter.

These are further classified into

1. Wall coated open tubular column(WOCT)
2. Support coated open tubular column(SCOT)
3. Porous-layer open tubular column(PLOT)

Normally capillary columns contain no packing and the stationary phase is coated directly inside the tubing. It cannot handle samples more than 0.1 μ l. Capillary columns are widely used for

1. Shorter retention time
2. Greater inertness
3. Longer life
4. Lower bleed
5. Higher efficiency
6. Greater reproducibility

5.4. LIQUID CHROMATOGRAPHY:

High pressure or high performance liquid chromatography (HPLC) is similar to gas chromatography in that the chemical components of a mixture are separated as the mixture is forced through a column packed with fine particles. In the gas chromatography the substance is carried through the column in vaporized form by an inert gas whereas in HPLC it is carried through the column in liquid form by the solvent. The main difference between these two is that there is no need to vaporize the samples in liquid chromatography but it has to be vaporized in gas chromatography. The liquid chromatography is more or less the same as HPLC but the difference is that HPLC is in need of high pressure for the separation of species; the liquid chromatography can be performed either in a column or an open bed but HPLC could be performed only in columns because the application of pressure on an open bed will not be effective. It is widely used of all the analytical separation processes. The reason for the popularity of this method is its high sensitivity, its ready adaptability to accurate quantitative determination, suitability for separating non-volatile species. Examples of such materials are amino acids, Proteins, hydrocarbons, carbohydrates, drugs, pesticides, antibiotics, steroids and a variety of inorganic substances.

The basic components are

1. Solvent reservoir for the mobile phase and its treatment.
2. Pumping system to force the liquid mobile phase through the column
3. Sample injection system for the introduction of sample in the mobile phase.
4. Chromatographic column with an appropriate length of stationary phase.
5. Detection System for the detection of sample components as they elute from the column
6. Microprocessor or recorder to provide a readable signal proportional in magnitude to the amount of each component present in the analyze.

The mobile phase liquid from the solvent reservoir is delivered to the column by some types of pump. The pumping system must be pulse free or a pulse damper must be installed to avoid instability in the detector. These are three types of pumps available they are

- Reciprocating piston pumps
- Syringe or displacement type pumps
- Pneumatic or constant pressure pumps

Sampling valves or loops are used to inject the sample into the flowing mobile phase. The sample dissolves in the mobile phase the different species of the sample are separated from each other while it passes 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.

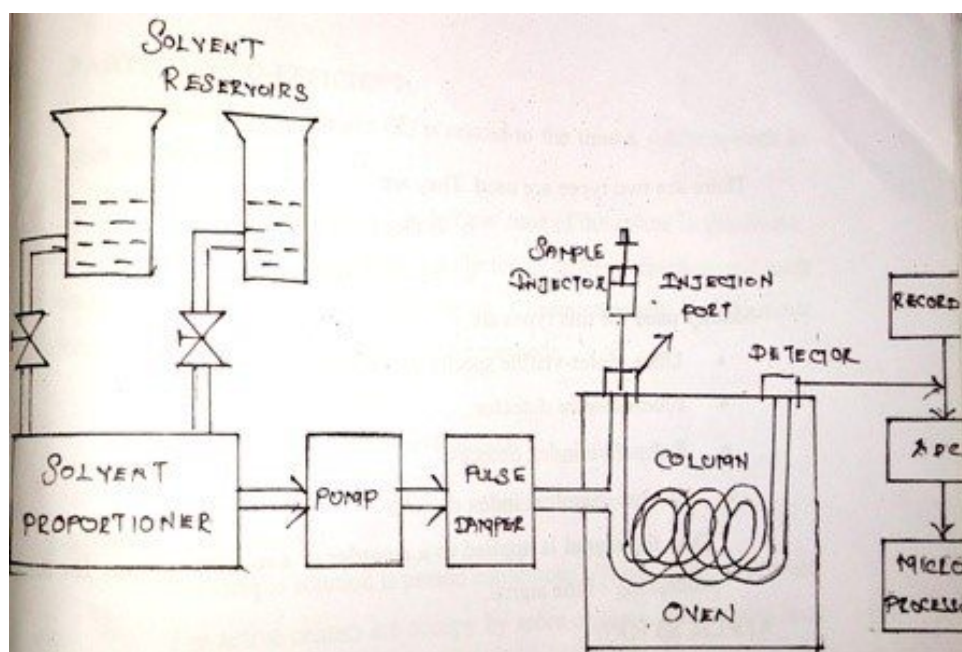


Fig: 5.4 LIQUID CHROMATOGRAPHY

These are two types are used. They are

- Bulk property detectors
- Solute property detectors

Mainly used for this types are

- Ultra violet-Visible spectrophotometric detector
- Fluorescence detector
- Refractive index detector
- Electro chemical index detector

Then the signal is applied to a recorder or a microprocessor for further processing of the signal

5.4.1. APPLICATION:

- It is applied in the process of isolation and purification of compounds.
- Chemical separation can be done by using liquid chromatography
- A particular compound could be identified with great accuracy.

5.5. RETENTION TIME:

The time it takes after sample injection for the analyze peak to reach the detector is called retention time.

5.6. DEAD TIME:

The time taken by an instrument to begin its response for a change in the measured quantity is called as dead time.

5.8. DETECTORS:

A Detector which is located at the exit of the separation column senses the presence of the individual components as they leave the column.

5.8.1. Principle of operation and construction of Thermal Conductivity Detector (TCD):

The thermal conductivity detector is a simple most widely used type of detector. It is based on the principle that all gases have the ability to conduct heat in varying degrees. This difference in heat conduction can be used to determine quantitatively the composition of a mixture of gases. It is also called kathrometer uses a heated filament as a sensing element which is placed in the path of emerging gas stream.

The heated element may be fine platinum, gold, tungsten wire or a semi conducting thermistor. The resistance of the wire or thermistor gives a measure of the thermal conductivity of the gas. In contrast to the wire detector the thermistor has a negative temperature co-efficient. The amount of heat from the filament by conduction to the detector will depends on the thermal conductivity of the gas.

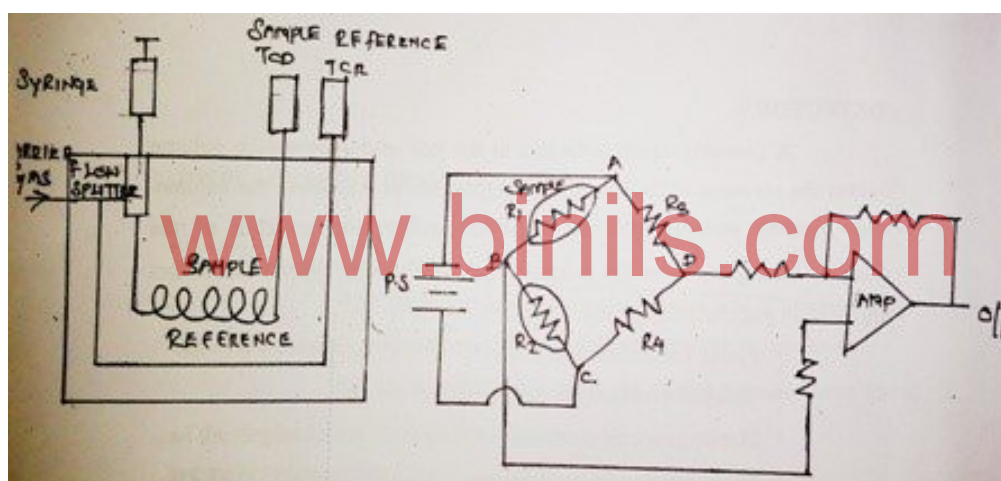


Fig: 5.5

The arrangement has two sensing filament in the Wheatstone bridge. One element is located in the effluent from the column and the other one in the gas stream ahead of the sample injection chamber. These elements are labeled as sample and reference as shown in above figure. The carrier gas stream is splitted into two, one carries the sample is the sample or column line and the other one carriers none is the reference line. In either case, the effect of thermal conductivity of the carrier gas is cancelled and the effects of vibration in flow rate, pressure and electrical power are minimized. The two temperature sensing elements are arranged in such a way to measure the change in resistance of R1(sample) with respect to resistance R2(ref), R3&R4 are constant. The output of the bridge is fed to the recorder through an amplifier to adjust the signal if it is greater than span rating of the recorder. For the balanced bridge condition, when the carrier gas alone flows through the two sensing element no current flows in between B and D.

$$R1/R2=R3/R4$$

The bridge is unbalanced due to the introduction of sample in the carrier gas line which increases the current flow in between B&D. the magnitude of the current is used to detect and measure the magnitude of the gas component vapour passing over the measuring cell.

5.8.2. ADVANTAGES:

- It is simple and large linear dynamic range.
- General response to both organic and inorganic species.

5.8.3. DISADVANTAGES:

- It is low sensitivity.

5.9. FLAME IONIZATION DETECTOR (FID):

It is the most widely used and generally applicable detector for gas chromatography. It responds with high sensitivity to all organic compounds. It adds hydrogen to the column effluent and the mixture is passed through a jet where it is mixed with external air and burned. The flame jet and a cylinder which is positioned above the tip of the flame forms the twin electrode. The flame jet is one electrode. The collector cylinder is other electrode. A potential of few hundred volts is applied across the burner tip and the collector electrode which is located above the flame. Most organic compounds when pyrolyzed at the temperature of a hydrogen/air flame produce ions and electrons.

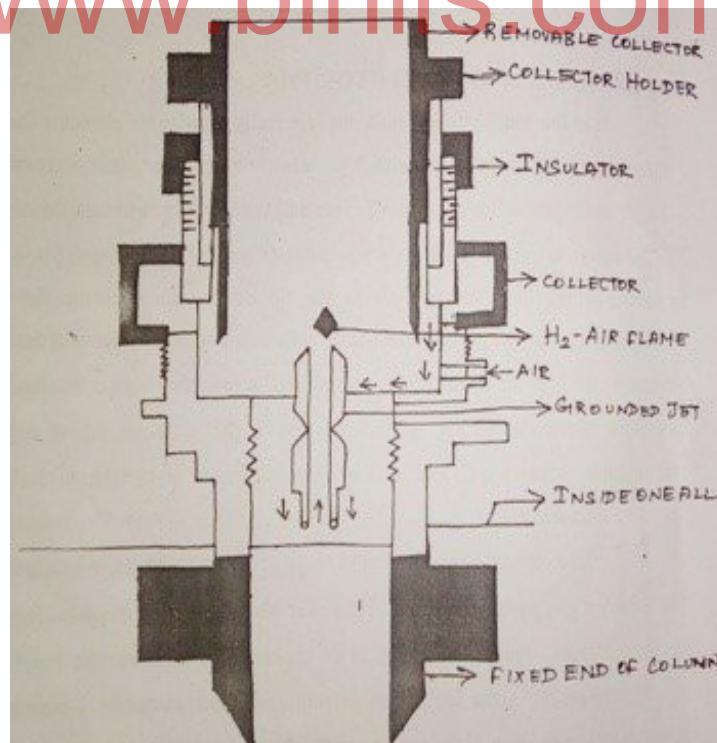


Fig: 5.6. FLAME IONIZATION DETECTOR

The current across the electrodes remains constant, when only the inert carrier gas passes the flame. However when ionized material from the column effluent enters the

flame, it is broken into ions by the hot flame. These ions result in the ionization current and there would be a consequent change in the current flowing across the electrodes. The magnitude of the variation in current would be directly proportional to the number of ions or electrons formed in the flame gases which in turn would be proportional to the carbon content of the organic molecules in it.

The current flowing through an external resistor is sensed as a voltage drop, then amplified and finally sent to an output device like a recorder or a microcomputer. Functional groups such as carbonyl, alcohol, halogen amine yield fewer ions or none at all in a flame.

In addition the detector is insensitive toward non-combustible gases such as H_2O , CO_2 , SO_2 and NO_x . These properties make the flame ionization detector a most useful general detector for the analysis a most organic samples including those are contaminated with water and oxides of nitrogen and sulfur. The insensitivity of the flame ionization detector to water makes it particularly useful for the detection of pollutants in natural water samples. The flame ionization detector is having the advantages of high sensitivity large linear response and low noise. It is generally rugged and easy to use but the disadvantage is that the emerging components get destroyed in flame.

5.10. FLAME PHOTOMETRIC DETECTOR (FPD):

Flame Photometric Detector is essentially special type of flame emission filter photometer used primarily for the determination of volatile Sulphur or phosphorous compounds. The column effluent passes into a dual Hydrogen enriched, lower temperature flame within a shield. Both air and hydrogen are supplied as makeup gases to the carrier gas. Only the upper flame is viewed. Phosphorous forms as HPO species that into band emissions at 510 and 526 nm around the sides and base of the flame. A sulphur compound emits a series of bands around 394nm but also overlapping the phosphorous spectrum. The detector responds to the concentration. These are about 100 times less sensitive to phosphorous than the thermionic emission detector.

5.11. ELECTRON CAPTURE DETECTOR (ECD):

The ECD works on the principle that the ionization current setup by certain radioactive sources like nickel-63(Ni_{63}) or tritium (H^3) absorbed in titanium or scandium gets reduced when an electron capturing compound is introduced into the cell. In effect ECD measures the loss of signal due to recombination phenomenon rather than measuring a positively produced electric current. Nickel 63 is a higher energy source the tritium and can be used up to $400^\circ C$

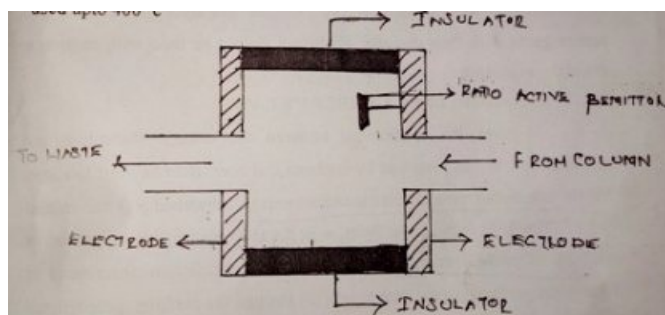


Fig: 5.7. ELECTRON CAPTURE DETECTOR

Although the sensitivity of the nickel detector is about 5 times less than that of a tritium unit, the standing current remains constant and the detector is much less susceptible to contamination.

5.11.1. WORKING:

The effluent from the column is allowed to pass over the β emitter (Ni^{63} or H^3). An electron from the emitter causes ionization of the carrier gas (mostly nitrogen carrier) and the production of a burst of electrons. In the absence of the organic species a constant standing current between a pair of electrodes of the organic species a constant standing current between a pair of electrodes results from this ionization process. The current decreases with the presence of organic molecules that tend to capture electrons. It is an sensitive to functional groups such as a mines, alcohols and hydrocarbons. The linearity of ECD is a function of the cell design and the composition of the carrier gas. The nitrogen and Hydrogen are the best carrier gases with their detector. Hydrogen should be used with caution to avoid the explosion.

5.12. EMR SPECTRUM:

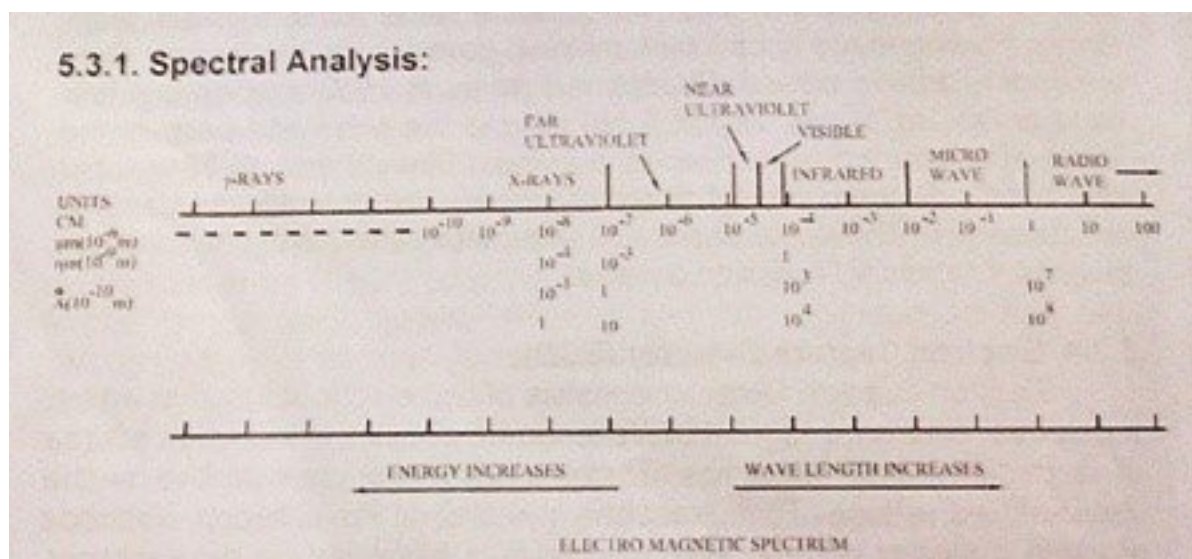


Fig: 5.8. EMR SPECTRUM

The most important of all instrumental method of analysis are methods based on the absorption of Electromagnetic radiation in the visible, Ultraviolet and infrared ranges. According to the quantum theory, the energy states of an atom or molecule are

defined and for any change from one state to another would, therefore require a definite amount of energy. If this energy is supplied from an external source of radiation, the exact quantity of energy required to bring about a change from one state to another will be provided by photons of one particular frequency, which may be selectively absorbed.

The interaction of matter and radiation takes place throughout the entire electromagnetic spectrum. The range of radiation extends from cosmic rays of wave length 10^{-9} nm to radio waves longer than 1000km. within these ranges are gamma rays, X rays, far, middle and near ultraviolet rays, the visible light portion, infra red and micro waves. The nature of all these radiations and wave length and in effect they can produce in matter. Molecules possess three types of internal energy:

- 1) Electronic.
- 2) Vibrational.
- 3) Rotational.

The chemical and physical effects of various types of radiation are quite different, and these differences can be understood in terms of the various energies of the photons.

- 1) In the radio frequency range, the energy of one photon is very low and the energy transitions are concerned with recitation of nuclear spin states of substances in a magnetic field.
- 2) In the microwave region, there are changes in electron spin status for substations with unpaired electronics when in a magnetic field.
- 3) In infra and ultra violet region, absorption causes in rotational and rotational-vibrational energy status.
- 4) In visible and ultra violet region, absorption causes changes of the valence electronics accompanied by rotational- vibrational energy changes.
- 5) X-rays cause the ejection of inner electronics from matter.
- 6) Gamma rays can cause changes in nucleuse.

5.13. BEER'S Law:

The relation between energy absorption and concentration is derived by beer and lambert and states that the amount of monochromatic radiation energy absorbed or transmitted by a solution is an exponential function of concentration of the absorbing substances present in the path of radiant energy. The relationship between radiant power and concentration can be derived, if the wave length and the distance traversed by the beam in the sample remain constant.

5.14. IR/UV Spectro photometer:

The infrared spectrometers are useful in analysis of almost all substances. These operate in the band (infrared) usually called heat radiation. It is usually found in petroleum refining and synthetic rubber production, where it is used for analysis of hydro-carbons and gases. It is also used in chemical plants for analysis of both organic and inorganic compounds and it the pharmaceutical work for determining the structure of penicillin etc.

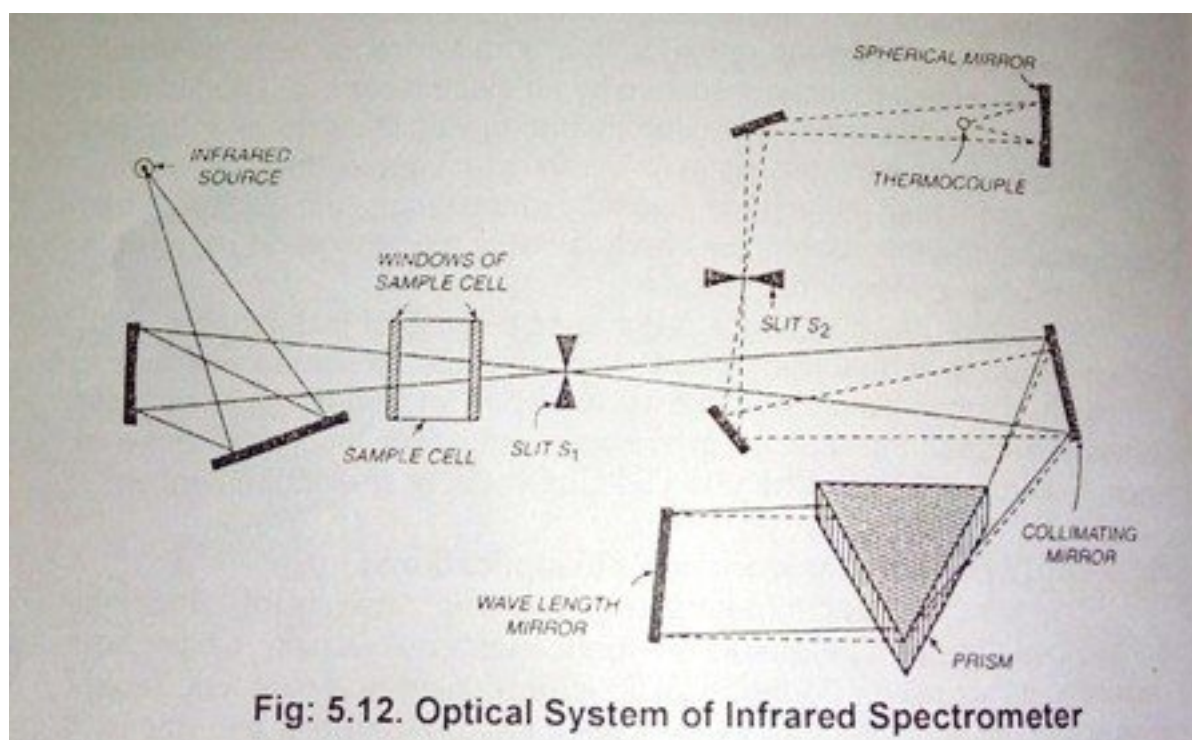


Fig: 5.12. Optical System of Infrared Spectrometer

The optical system of a typical infrared spectrometer, which is very commonly used, is shown in the figure the infrared radiation beam provided by an electrically heated resistor passes to a mirror then to a parabolic mirror and after passing through the sample cell, (containing the unknown substances), falls on slit s1 which serves to form a beam of very narrow width. The beam then passes to a collimating mirror, where it is rendered parallel, and then through the prism where it is refracted. The beam is next reflected by the collimating mirror to a plane mirror and then to slit s2. The beam is finally reflected to the thermocouple, where the beam intensity is measured.

The wavelength of the infrared radiation which is passed to the thermometer is determined by the angle of setting of the wavelength mirror. As this mirror reflects the refracted beam and governs which part of the beam falls on slit s2, it is possible to scan the wavelength spectrum by slowly rotating the wavelength mirror.

This thermocouple will measure the intensity of the beam at each wavelength. The effect of temperature is accounted for by employing a temperature compensator which positions the mirror accordingly. The compensator consists of a bimetallic strip and it corrects for the temperature co-efficient of refractive index of the prism. Generally the prisms of four different types of materials are available and their selection depends upon the wavelength range most adaptable to the substance under analysis. The spectrographic data are recorded by an ordinary automatic balance potentiometer.

Differential analysis is sometimes employed particularly for analysis of slight impurities or slight variations from desired concentrations. In this method, it is compared to the transmission of a pure substance contained in the compensating cell.

The concentration of various components of the sample substance can be determined from the amount of absorption at any wavelength, by the Beer's law, according to which

$$C = \frac{1}{ax} \log_{10} \frac{I_0}{I_x}$$

C = Concentration of substance

a = absorption factor of substance

x = thickness of sample along optical path

I_0 = intensity of beam before sample

I_x = intensity of beam after sample

Here a, x and I_0 are constant and can be found by a trial calibration of the spectrometer may be determined at any one wavelength in the spectrogram.

It may be noted from the Beer's law that it is necessary that the analysis be made by studying a complete spectrogram, but it is possible to perform continuous analysis by operating the spectrometer at one wavelength. This wavelength can be so selected that the particular analysis made is the most sensitive. Based on this is given in fig, the arrangement for the analysis of a gas flowing continuously through a sample cell. In this method, the gas absorbs at a particular wavelength, which is passed by the upper filter but not by the lower filter. The amount of radiation obtained by each bolometer then depends on the concentration of the absorbing gas.

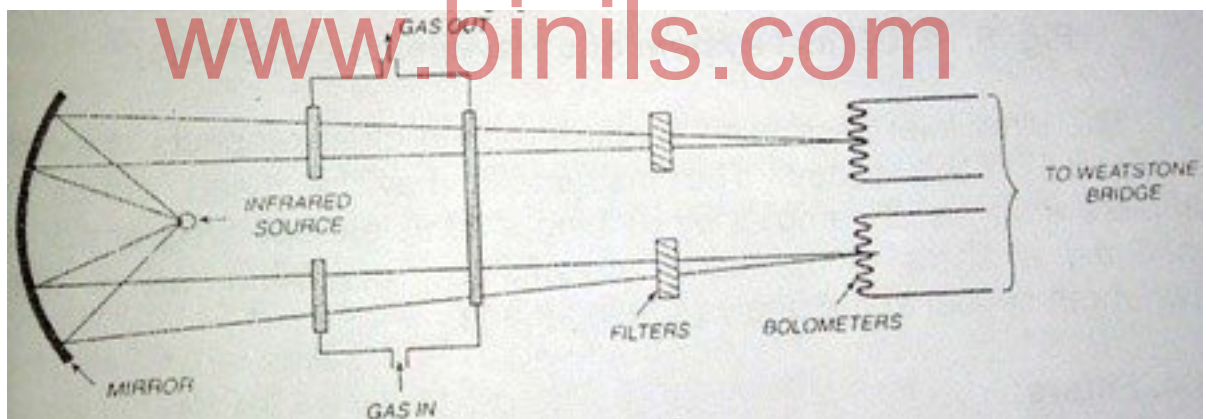


Fig : 5.13.

This method is very simple and is used for a wide variety of gases except oxygen, hydrogen and nitrogen which do not in the infrared region. Such devices can also be employed for analysis of liquid.

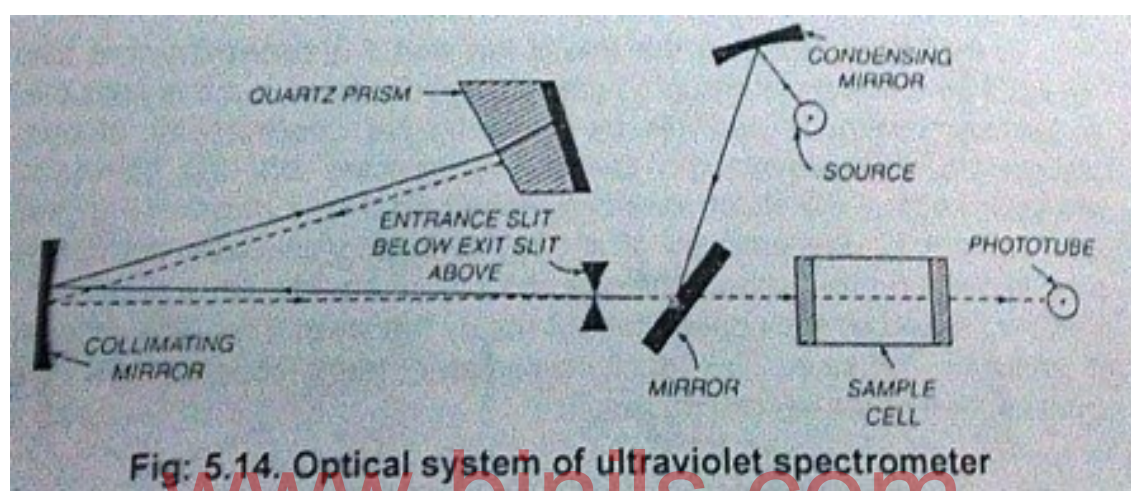
5.14. Ultraviolet Absorption Spectrometers:

These are widely used for nearly all types of analysis indicated, these operate in the range from 0.2- 0.8 micron. The optical path of a widely used ultraviolet spectrometer (photoelectric quartz spectrometer) is shown in fig. An image of the light source (tungsten lamp for use in the visible region from 0.3-1.0 microns and hydrogen-discharge lamp for use in the ultraviolet region from 0.22-0.35 micron) is focused by the condensing mirror and by the flat mirror on the entrance slit, from where it proceeds to a collimating mirror and through a quartz prism and the collimating mirror, the portion

of the spectrum passes through the exit slit and then through the sample cell falls on the phototube.

The intensity of the beam is measured by a suitable amplifier connected to an automatic balance potentiometer instrument. The particular wavelength passed through the sample cell falls on the phototube.

The intensity of the beam is measured by a suitable amplifier connected to an automatic balance potentiometer instrument. The particular wavelength passed through the sample cell determined by the angle of setting of the prism. The angle of the quartz prism determines which part of the refracted beam falls on the exit slit. The sample cell and be arranged for continuous flow of gas and liquid samples.



The ultraviolet absorption spectroscopy in other respects is similar to infrared spectroscopy. The instrument may be used either for complete analysis of samples by running a complete spectrogram or for continues analysis of process streams by operating at a fixed wavelength critical to the analysis being made.

5.15. Range of IR/UV radiation:

Infrared radiation extends from the nominal red edge of the visible spectrum at 700 nanometers (nm) to 1 mm. This range of wavelengths corresponds to a frequency range of approximately 430 THz down to 300 GHz. Below infrared is the microwave portion of the electromagnetic spectrum.

Ultraviolet (UV) is an electromagnetic radiation with a wavelength from 10 nm (30 PHz) to 400 nm (750 THz), shorter than that of visible light but longer than X-rays. UV radiation is present in sunlight. It is also produced by electric arcs and specialized lights such as mercury-vapor lamps, tanning lamps, and black lights. Although lacking the energy to ionize atoms, long-wavelength ultraviolet radiation can cause chemical reactions and causes many substances to glow or fluoresce. Consequently, biological effects of UV are greater than simple heating effects, and many practical applications of UV radiation derive from its interactions with organic molecules.

5.16. Measurement of IR/UV radiation:

The intensity of UV radiation is measured in the units of milliwatts per square centimeter (mW/cm^2) which is energy per square centimeter received per second. Also, it is measured in the units of millijoules per square centimeter (mJ/cm^2), which is energy received per unit area in a given time.

A variety of instruments are commercially available for measuring UV radiation in the laboratory and in the workplace. Specifications and purchasing information can be obtained from suppliers of workplace monitoring equipment.

An infrared thermometer is a thermometer which infers temperature from a portion of the thermal radiation sometimes called blackbody radiation emitted by the object being measured. They are sometimes called laser thermometers as a laser is used to help aim the thermometer, or non-contact thermometers or temperature guns, to describe the device's ability to measure temperature from a distance. By knowing the amount of infrared energy emitted by the object and its emissivity, the object's temperature can often be determined. Infrared thermometers are a subset of devices known as "thermal radiation thermometers".

5.17. IR/UV radiation sources:

Gas lasers, laser diodes and solid-state lasers can be manufactured to emit ultraviolet rays, and lasers are available which cover the entire UV range. The nitrogen gas laser uses electronic excitation of nitrogen molecules to emit a beam that is mostly UV. The strongest ultraviolet lines are at 337.1 nm and 357.6 nm, wavelength. Another type of high power gas laser is the excimer laser. They are widely used lasers emitting in ultraviolet and vacuum ultraviolet wavelength ranges. Presently, UV argon-fluoride (ArF) excimer lasers operating at 193 nm are routinely used in integrated circuit production by photolithography. The current wavelength limit of production of coherent UV is about 126 nm, characteristic of the Ar_2^* Excimer laser.

Direct UV-emitting laser diodes are available at 375 nm. UV diode lasers have been demonstrated using Ce:LiSAF crystals (cerium-doped lithium strontium aluminum fluoride), a process developed in the 1990s at Lawrence Livermore National Laboratory. Wavelengths shorter than 325 nm are commercially generated in diode-pumped solid-state lasers. Ultraviolet lasers can also be made by applying frequency conversion to lower-frequency lasers.

Ultraviolet lasers have applications in industry (laser engraving), medicine (dermatology, and keratectomy), chemistry (MALDI), free air secure communications, computing (optical storage) and manufacture of integrated circuits.

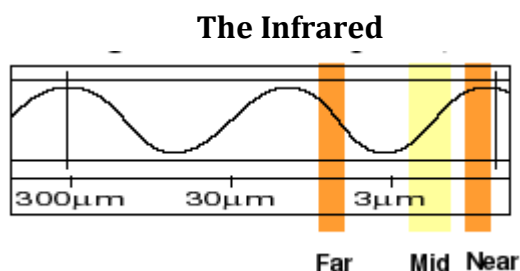


Fig: 5.15. Infrared Region of Electromagnetic Spectrum

Infrared light lies between the visible and microwave portions of the electromagnetic spectrum. Infrared light has a range of wavelengths, just like visible light has wavelengths that range from red light to violet. "Near infrared" light is closest in wavelength to visible light and "far infrared" is closer to the microwave region of the electromagnetic spectrum. The longer, far infrared wavelengths are about the size of a pin head and the shorter, near infrared ones are the size of cells, or are microscopic.

5.18. Monochromator:

Monochromators are the optical systems, which provide better isolation of spectral energy than the optical filters and are therefore preferred where it is required to isolate narrow bands of radiant energy. Monochromators usually incorporate a small glass of quartz prism or diffraction grating systems as dispersing media. The radiation from the light source is passed either directly or by means of a lens or mirror into the narrow slit of the monochromator and allowed to fall on the dispersing medium, where it gets isolated. The efficiency of such monochromators is much better than that of filters.

Isolation of different wavelength in a prism monochromator depends upon the fact that the refractive index of materials is different for radiation of different wavelengths. If a parallel beam of radiation on a prism, the radiation of two different wavelengths will be bent through different angles the greater the difference between these angles, the easier is to isolate the two wavelengths.

Light from the source S is made into a parallel beam and made to fall on a prism after it is passed through entrance slit s_1 and mirror m_1 . The entrance slit is at the focus of mirror m_1 . The prism disperses the light and photons of different wavelengths are deflected at different angles. If the dispersed beam is again refocused, the focal point for photons of one wavelength passes through the slit the other wavelength is blocked. There are several ways of selecting a particular wavelength. It may be chosen by local selection with movable exit slits, or the prism is moved to shift the spectrum by keeping the slits fixed.

5.19. Sample Handling:

Liquid may be contained in a cell or cuvette made of transparent material such as silica, glass or perspex. The faces of these cells through which the radiation passes are highly polished to keep reflection and scatter losses to a minimum and the faces should be parallel and placed perpendicular to the beam of radiation. Solid samples are not suitable for direct photometry. It is usual to dissolve the solid in a transparent liquid.

Gases may be contained in cells which are sealed to make them air tight. A sample holder is generally placed between the light source and the detector.

Sample and reference cells must be matched to obtain accurate results. The cells must be cleaned reference by nitric or aqua regia before and after each use. After cleaning, it should be thoroughly rinsed and dried. The path length may be changed if it is dried in oven or in flames. The faces of the cell should not be handled without gloves, finger prints may cause appreciable changes in transmittance of the cell.

Normally 10mm path length rectangular cells are used for ultra violet and visible regions. In infrared region cells of 0.1-1 mm path lengths are used. Densely coloured samples and turbid solutions may require shorter path lengths, which can be obtained by inserting transparent inserts like silica spaces in the standard cell. This demountable cell is very useful in cleaning the contaminants which are difficult to remove in normal cleaning procedures. The cylindrical cells are cheap but it is difficult to position the sample cell.

Review Questions: Unit-V

PART-A

1. What is Chromatography?
2. What is Retention time?
3. What is Dead time?
4. Explain TCD?
5. Expand FPD?
6. What are the types of Chromatography?
7. Define Sample Cell?
8. Define monochromators?
9. Give the expansion of IR/UV.

PART-B

1. Explain the principle of Gas chromatography?
2. What is Detectors?
3. Write the applications of Liquid Chromatography?
4. What are the advantages of Chromatography?
5. What is Beer's Law?
6. What is meant by Spectral Analysis?

PART-C

1. With a neat sketch explain the operation of Liquid Chromatography?
2. Explain the operation and construction of TCD?
3. Explain the operation of Gas chromatography with a neat sketch?
4. With neat diagram, explain the construction and operation of FID?
5. With neat diagram, explain the operation of FPD?
6. With neat diagram, explain the operation of ECD?
7. Explain the working and applications of IR/UV Photometer with neat diagram?
