

INTRODUCTION TO ANALYTICAL INSTRUMENTATION

Multiple Choice Type Questions

1. In heat of combustion method, H₂ is supplied at pressure of about

[WBUT 2009]

- a) 1.5 – 2 kg/cm²
- b) 3 – 3.5 kg/cm²
- c) 4 – 6 kg/cm²
- d) above 10 kg/cm²

Answer: (a)

2. The analyzer cell where a third electrode may be added to increase life of the cell is

[WBUT 2009, 2018]

- a) Zirconia fuel cell
- b) Palarographic cell
- c) Hot wire TCD analyzer
- d) Hersh Cell

Answer: (d)

3. Ilkovic equation appears in the electrochemical analysis method of

[WBUT 2009]

- a) amperometric titration
- b) voltammetry
- c) coulometry
- d) electrogravimetry

Answer: (b)

4. Which viscometer is used for both of Newtonian and Non-Newtonian fluid?

[WBUT 2009, 2012, 2015]

- a) Saybolt's viscometer
- b) Ostwald viscometer
- c) cone and plate viscometer
- d) none of these

Answer: (d)

5. Dew point is expressed as

[WBUT 2009, 2012, 2013, 2014, 2018]

- a) % (percentage)
- b) °C

c) V_{ppm} d) none of these

Answer: (b)

6. In Flame ionization detector, the magnitude of current is proportional to

[WBUT 2009, 2012]

- a) proton number
- b) neutron number
- c) effective carbon number
- d) mass number

Answer: (c)

7. Aerosol is formed by

[WBUT 2009, 2012, 2015, 2018]

- a) Bolometer
- b) Scintillation counter
- c) Nebulizer
- d) Nephelometer

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Answer: (c)

8. In Nernst equation of Zirconia O₂ analyzer, value of n is taken as

[WBUT 2009, 2014, 2017, 2018]

- a) 0
- b) 1
- c) 3
- d) 4

Answer: (d)

9. Pyroelectric detector is formed temperature sensitive

[WBUT 2009, 2018]

- a) Resister
- b) Inductor
- c) Capacitor
- d) Diode

Answer: (c)

10. The temperature range of inductively coupled plasma is

[WBUT 2009, 2017]

- a) 4000 – 5000 K
- b) 6000 – 7000 K
- c) 7000 – 8000 K
- d) 13000 – 14000 K

Answer: (b)

11. The definition of specific gravity (for solid) is the ratio of

[WBUT 2010, 2013]

- a) density of solid and density of reference solid
- b) density of solid and density of water
- c) density of solid and density of gas
- d) none of these

Answer: (b)

12. Measurement of viscosity involves measuring

[WBUT 2010, 2012, 2014, 2017, 2018]

- a) Frictional force
- b) Coriolis force
- c) Centrifugal force
- d) Buoyant force

Answer: (a)

13. Thin film of which of the following is suitable for moisture measurement?

[WBUT 2010]

- a) titanium oxide
- b) tantalum oxide
- c) tin oxide
- d) tellurium oxide

Answer: (a)

14. In a Newtonian fluid, viscosity does not depend on

[WBUT 2010]

- a) pressure
- b) temperature
- c) flow rate

d) all of these

Answer: (c)

15. By 'heat of reaction' method, gas can be estimated upto

[WBUT 2010, 2014]

- a) 1%
- b) 0.1%
- c) 0.01%
- d) 0.001%

Answer: (a)

16. Kinematic viscosity of a liquid depends on [WBUT 2011]
a) volume of the liquid
b) ambient temperature
c) density of the liquid
d) all of these
- Answer: (d)
17. Which material cannot be used as a hygroscopic coating for measurement of moisture? [WBUT 2011, 2014]
a) silver chloride
b) lithium chloride
c) zinc chloride
d) phosphoric acid
- Answer: (d)
18. Typical detector of an ultraviolet gas analyzer is [WBUT 2011, 2012]
a) thermistor
b) thermocouple
c) thermal conductivity detector
d) none of these
- Answer: (d)
19. Density can be measured by using a / an [WBUT 2013]
a) hygrometer
b) hydrometer
c) anemometer
d) tachometer
- Answer: (a)
20. The Zirconia fuel cell is used to determine the [WBUT 2013]
a) density of a fluid
b) oxidation-reduction potential of an electrolyte
c) moisture content of a gas
d) percentage of oxygen in a gas mixture
- Answer: (d)
21. Katharometer cell is used to measure the [WBUT 2013, 2017]
a) pH of liquid
b) conductivity of liquid
c) thermal conductivity of gas
d) potential difference
- Answer: (c)
22. Which of the following instruments is useful of oxygen analysis? [WBUT 2014]
a) paramagnetic analyzer
b) gas chromatography
c) flame ionization detector
d) heat of reaction analyzer
- Answer: (a)
23. Using the terms "oxidized" and "reduced" the potential of an electrode is given by [WBUT 2016]
- $$E = E_0 - \frac{0.0591}{n} \log \frac{[\text{oxidized state}]}{[\text{reduced state}]}$$
- originally derived by
- a) Daimler b) Einstein c) Nernst d) none of these

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Answer: (c)

24. In a dairy industry, which of the following units is preferred for specific gravity measurement? [WBUT 2017]

- a) Twaddell b) Ba c) Quevenne d) API

Answer: (c)

25. Which of the following microwave bands is suitable for moisture measurement? [WBUT 2017]

- a) S band b) X band
c) Both S band and X band d) K band

Answer: (d)

[Water molecules attenuate in the *S* and *X bands* of the *microwave* radiation, in *K band* they produce molecular resonance which is a unique]

26. By heat of reaction method, gas can be estimated up to

- a) 1% b) 0.1% c) 0.01%

[WBUT 2017]

- d) 0.001%

Answer: (a)

27. Gow-Mac densitometer is used to measure the density of

- a) solid b) liquid c) gas

[WBUT 2017]

- d) plasma

Answer: (b)

28. Vibrating densitometer is used for density measurement of [WBUT 2018]

- a) both solids & gases b) both solids & liquids
c) both liquids & gases d) solids, liquids, gases

Answer: (b)

29. Which viscometer is used for both Newtonian and non-Newtonian fluid?

[WBUT 2018]

- a) Saybolt viscometer b) Ostwald viscometer
c) Cone and plate viscometer d) Capillary viscometer

Answer: (d)

30. Which of the following microwave bands is suitable for moisture measurement? [WBUT 2018]

- a) S band b) X band
c) Both S band and X band d) K band

Answer: (b)

31. In sugar industry, which of the following units is preferred for specific gravity measurement? [WBUT 2018]

- a) Baume b) API c) Twaddle

- d) Quevane

Answer: (b)

Short Answer Type Questions

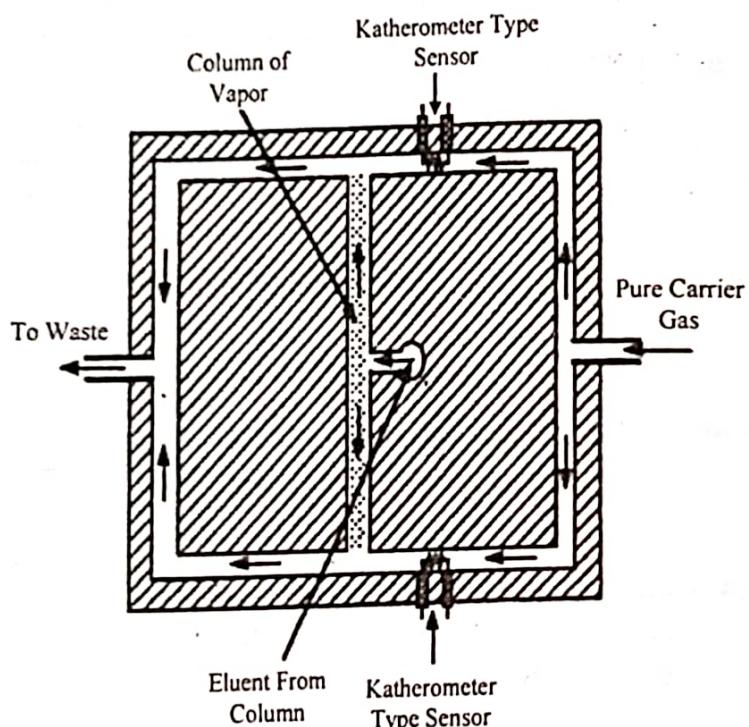
1. Describe the operation of the Gow-Mac gas density detector. [WBUT 2010, 2014]
OR,

Explain the operation of Gow-Mac density measurement technique. [WBUT 2016]

Answer:

GOW-MAC Gas Density Detector

A typical diagram of the GOW-MAC gas density balance is shown in figure below.



The GOW-MAC Gas Density Balance

The sensor consists of a bridge of tubes with three vertical tubes all connected by horizontal tubes at the top and the bottom. Pure carrier gas enters at the center of the right hand vertical tube and splits into two streams one passing along the lower horizontal tube and the other along the upper horizontal tube. The eluent from the column enters the center of the middle tube and the flow also splits into two streams and each meets the respective flow from the right-hand tube. The flows in the two horizontal tubes finally pass up and down the left-hand vertical tube to meet at the center and then exit to waste. Flow sensors are situated in the horizontal tubes between the right-hand vertical tube and the center vertical tube. When only carrier gas is present in the system, the horizontal flows equal and the temperature and thus the potential across the filaments of the two sensors are the same. When a solute is eluted from the column, vapor will be present in the center vertical tube and the pressure at the top and bottom of the tube will differ. This will result in a differential flow through the horizontal tubes with a consequent change in the output from the sensors. As the differential flow will be proportional to the pressure difference between the right-hand column of pure carrier gas and the center column full

of vapor, the output from the sensor filaments will be proportional to the **vapor density of the solute and consequently** be related to the **molecular weight**. In fact with a second detector that measured the concentration of the solute, the gas density balance can be used to determine molecular weight of an eluted solute.

This device works well, has about the same sensitivity and linearity as the katherometer but, unfortunately, is no longer manufactured. It was one of the very few simple and inexpensive methods available for measuring the molecular weight of an elutes solute.

2. If λ_1, λ_2 and λ_m are the thermal conductivities of two gases and their mixture respectively, then explain how you can find out the percentage of each gas in the mixture.

Which type of gases can be analyzed by the heat of reaction method? [WBUT 2010]

Answer:

1st Part: Refer to Question No. 1(b) of Long Answer Type Questions.

2nd Part:

The sample at a percentage of combustible gases in mixture can be analyzed by heat of reaction method.

3. Define Newtonian and non-Newtonian liquids and state their difference.

[WBUT 2012]

Answer:

Newtonian fluids **obey Newton's laws** but non Newtonian fluids **does not obey Newton's laws**.

In a Newtonian fluid, the relation between the shear stress and the strain rate is **linear**, the constant of proportionality being the coefficient of viscosity. In simple terms, the size of the drops is directly related to the thickness of the fluid, all else being equal. In a non-Newtonian fluid, the relation between the shear stress and the strain rate is **nonlinear, and can even be time-dependent**. Therefore a **constant coefficient of viscosity** can not be defined. Multi-viscosity motor oil, which changes viscosity with temperature, is a common example.

Water is an example of a Newtonian fluid. Non-Newtonian fluids include catsup, paint, liquid detergent, liquid polymers and a variety of other liquids.

4. Describe with neat sketch, the constructional features and the principle of operation of a mechanical type paramagnetic oxygen analyzer. [WBUT 2012]

Answer:

Deflection-type oxygen analyser: It is First introduced by the legendary chemist Linus Pauling and his co-workers in 1946, this paramagnetic oxygen analyser is schematically shown in Fig. (i).

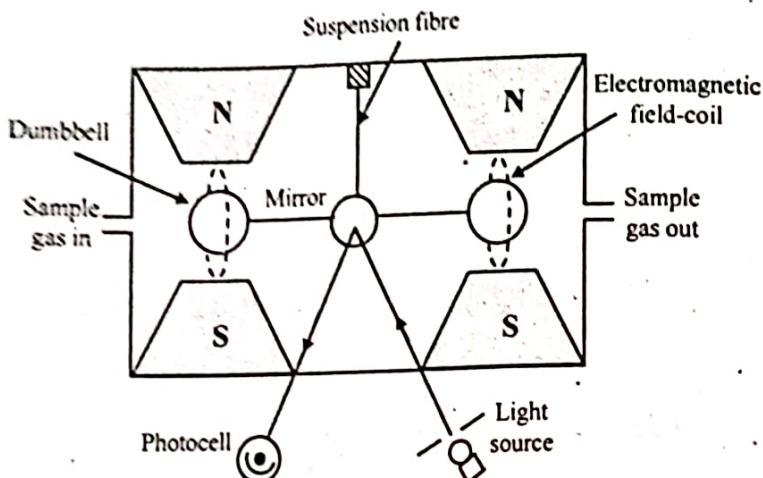


Fig: (i) Deflection-type paramagnetic oxygen analyser

It consists of a glass dumbbell **suspended by quartz fibre** between the poles of a permanent magnet. The dumbbell is filled with nitrogen or some other gas of **low magnetic susceptibility**. Magnetic pole pieces are in the shape of a wedge so that the field between them is non-uniform. Without the sample gas, the dumbbell stays slightly off from the strongest part of the magnetic field because of its nitrogen content. As the sample gas is let in, its oxygen content occupies the strongest part of the magnetic field and thus displaces the dumbbell further to cause a deflection of the light incident on the mirror in the suspension fibre. The force F that will be acting on each dumbbell is

$$F = K(\chi - \chi_d)$$

where χ is the magnetic susceptibility of the **sample gas**

χ_d is the magnetic susceptibility of the **material and content of the dumbbell**

K is a function of the **magnetic field and its gradient**.

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

Though sensitive, the instrument is delicate and it has to be installed on a vibration-free pedestal. Also, the sample gas needs to be filtered because dirt in the sample is likely to cause problems.

5. Define the following terms:

[WBUT 2013]

Relative humidity, viscosity, Psychrometer, moisture, partial pressure.

Answer:

Relative humidity:

This is the **ratio of moisture content of the gas to the maximum moisture the gas can contain at that temperature**. This is more precisely defined as the ratio of the **partial pressure of the vapour in the gas to its saturated partial pressure**. For an ideal gas, this is the ratio of the density of the vapour (ρ_H) to that at saturation (ρ_s),

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$$H_g = \rho_H / \rho_s$$

Viscosity:

Viscosity is a measure of the fluidity of the liquid or the gas. Many fluids undergo continuous deformation with the application of shearing stress. Application of a shear force, therefore, produces a flow. If this force-flow relation is linear, the fluid is Newtonian. For non-Newtonian fluids, the force flow relation is not only non-linear but changes from material to material. When continuous deformation occurs, the fluid tries to oppose this with a frictional resistance. This resistance can be measured in terms of consistency. Consistency of Newtonian fluids is called viscosity. Viscosity is typically different for different grades in a single process depending on the composition and is, therefore a convenient property to control product gradation.

Psychrometer:

A psychrometer is a device which is used for measuring dry bulb and wet bulb temperatures simultaneously. The psychrometer has two temperature sensors, one exposed to the atmosphere and one enclosed in a wet wick. Air is blown across the sensors, which causes evaporation and a reduction in temperature in the wet sensor. The temperature difference between the sensors is related to the humidity level. The lowest measurement uncertainty attainable is $\pm 4\%$.

The humidity measurement methods mostly measure relative humidity. The classical method is the dry and wet bulb system also known as psychrometer.

Moisture:

Moisture is often loosely defined as the amount of water absorbed or absorbed by a solid or a liquid. The standard method of moisture determination is by weighing. It is sometimes known as the gravimetric method and is used for calibrating other types of instruments. The sample is weighed when wet and subsequently when dry by driving off the moisture by desiccation. Percent moisture content is then calculated from

$$M_p = \left[\left(w_{\text{wet}} - w_{\text{dry}} \right) / w_{\text{wet}} \right] \times 100\%.$$

Partial Pressure:

In a mixture of gases, each gas contributes to the total pressure of the mixture. This contribution is the partial pressure. The partial pressure is the pressure the gas would exert if it were in the same volume and temperature by itself.

6. What is thermal conductivity? Deduce the expression for sensitivity of thermal conductivity gas analyser.
[WBUT 2013]

Answer:

Thermal conductivity is the property of a material to conduct heat. It is evaluated primarily in terms of Fourier's Law for heat conduction.

Heat transfer occurs at a higher rate across materials of high thermal conductivity than across materials of low thermal conductivity. Correspondingly materials of high thermal conductivity are widely used in heat sink applications and materials of low thermal conductivity are used as thermal insulation. Thermal conductivity of materials is temperature dependent. The reciprocal of thermal conductivity is called thermal resistivity.

Thermal Conductivity Gas Analysers

From the kinetic theory of gases, the thermal conductivity of a gas can be shown to be independent of pressure. This is true over a large range of moderate pressure. However, at very high and very low pressures, this is no longer valid. Thermal conductivity is a function of temperature and the functional relationship is given by

$$K_T = K_0 \left[a + \frac{273}{b} + T \right] \left(\frac{T}{273} \right)^{3/2}$$

where, K_T, K_0 = thermal conductivity at T K and 0 K respectively

T = temperature, K

a, b = constant

The thermal conductivity of a binary mixture is a function of the thermal conductivities of its two constituents and is given by an equation of the form

$$K = \frac{K_1}{1 + a \left(\frac{1 - x_1}{x_1} \right)} + \frac{K_2}{1 + b \left(\frac{x_1}{1 - x_1} \right)}$$

where, K, K_1, K_2 = thermal conductivity of the mixture, component 1 and component 2 respectively

x_1 = mole fraction of component 1

a, b = constants known as Wasiljewa constants.

This relation between K, K_1 and K_2 is complex and hence, mixture thermal conductivity is obtained by calibration at known mole fractions.

7. Define 'dew point'. Describe with a diagram the method of determining the relative humidity from a measurement of dew point. [WBUT 2017]

Answer:

1st Part: Refer to Question No. 2(d) (2nd part) of Long Answer Type Questions.

2nd Part:

The basic structure of the sensor unit for a chilled-mirror dewpoint hygrometer for measuring relative humidity is shown in figure below. The sample air is drawn to the metallic mirror surface through piping to determine the dew point temperature. As the mirror cools, condensation forms when its surface temperature falls below the dew point temperature, but evaporates and disappears at higher temperatures. The temperature of the metallic mirror when condensation form is measured using a platinum resistance thermometer, and the result is taken as the dew point temperature. Condensation conditions are monitored using a photo-detector with the reflection of a light-emitting diode (LED) on the mirror. Irradiated light is scattered when condensation is present, and the amount of reflected light changes with the mirror's surface condition. A peltier element is used to control the mirror's temperature.

8. Briefly explain the working principle of hair hygrometer. Indicate RH range and sources of error. [WBUT 2017]

Answer:

Principle of measurement and structure: The hair hygrometer uses the characteristic of the hair that its length expands or shrinks response to the relative humidity. The dimensions of various organic materials vary with their moisture content. A humidity change takes an effect on the moisture content in such materials. The length of human hair from which liquid are removed increases by 2 to 2.5% when relative humidity changes by 0 to 100%. Different types of human hair show different changes in length. However, there is still a relationship between the length of hair and relative humidity.

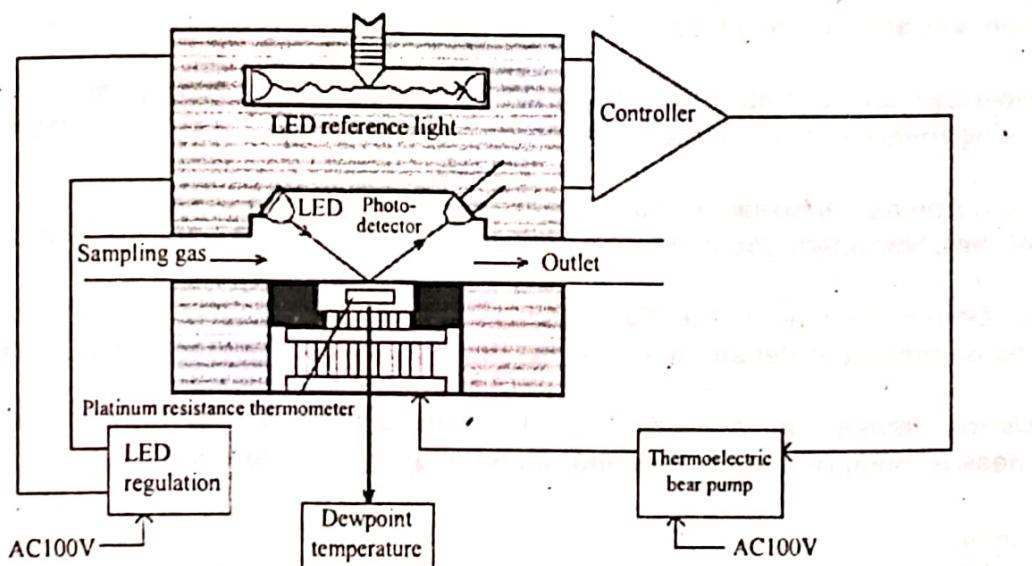


Fig: Chilled-mirror dewpoint relative humidity meter

RH Range

The length of human hair from which liquid are removed increases by 2 to 2.5% when relative humidity changes by 0 to 100%.

Sources of errors

1. The response of hair to humidity has hysteresis. The hair length changes more when humidity increases than when it decreases. The change of hair length observed when humidity increases is up to 5 to 6% larger than that observed when humidity decreases.
2. Hair is highly sensitive to contamination such as dust, ammonia, oil that adheres to hair.
3. The response time of the hair hygrometer depends on air temperature.
4. If a hair hygrometer is left in the low humidity condition for a long time, its reading changes causing large errors.
5. The response of hair to humidity has hysteresis. The hair length changes more when humidity increases than when it decreases.

9. Define Newtonian liquid and Non-Newtonian liquid.

[WBUT 2018]

Answer: Refer to Question No. 3(a) of Long Answer Type Questions.

Long Answer Type Questions

1. a) Describe the moisture measurement technique with necessary diagram.

[WBUT 2009]

OR,

Describe the moisture measurement technique with proper diagram. [WBUT 2018]

b) How may thermal conductivity gas analyzer be used to measure the concentration of the component gas of a binary gas mixture?

[WBUT 2009]

OR

How can concentration of the known component gas of binary mixture be found using thermal conductivity gas analyzer?

[WBUT 2015]

OR,

Describe how thermal conductivity analyser can be used to measure concentration of the component gas of a binary gas mixture.

[WBUT 2018]

c) Define density of the fluid. Describe with neat diagram the techniques for measurement of density of a process fluid for controlling the quality.

[WBUT 2009]

OR,

Define density of a fluid. Describe with a neat diagram the technique for measurement of density for controlling the quality of a process fluid.

[WBUT 2018]

Answer:

a) **Moisture Measurement:** A hygrometer is an instrument used for measuring the moisture content in the environment. Humidity measurement instruments usually rely on measurements of some other quantity such as temperature, pressure, mass or a mechanical or electrical change in a substance as moisture is absorbed. By calibration and calculation, these measured quantities can lead to a measurement of humidity.

Piezoelectric Hygrometer is a typical type of hygrometer used for moisture measurement.

A piezoelectric crystal, when excited by an ac signal, starts vibrating at a frequency which is the natural frequency of vibration of the crystal. This natural frequency of vibration is related to the mass of the crystal. Thus, if a piezoelectric crystal, can be somehow loaded with the moisture or water vapour of the atmosphere, its frequency of vibration will change. This property is utilized in the construction of piezoelectric hygrometer.

A suitable piezoelectric crystal, such as quartz, is coated with a hygroscopic material and exposed to the sample gas. The hygroscopic material absorbs the sample moisture, increases the mass of the vibrating piezoelectric crystal and thereby decreases its frequency of vibration. This decrease in the frequency of vibration is a measure of the moisture content of the sample.

To increase the accuracy of measurement, two identical piezoelectric transducers may be used – one exposed to the moist sample and the other to dry gas – at the same flow rate. A little later, the gas flow between transducers may be interchanged by energizing and de-energizing the solenoid valves that are suitably placed in the flow path of gases. This ensures a better accuracy and maintenance of transducers.

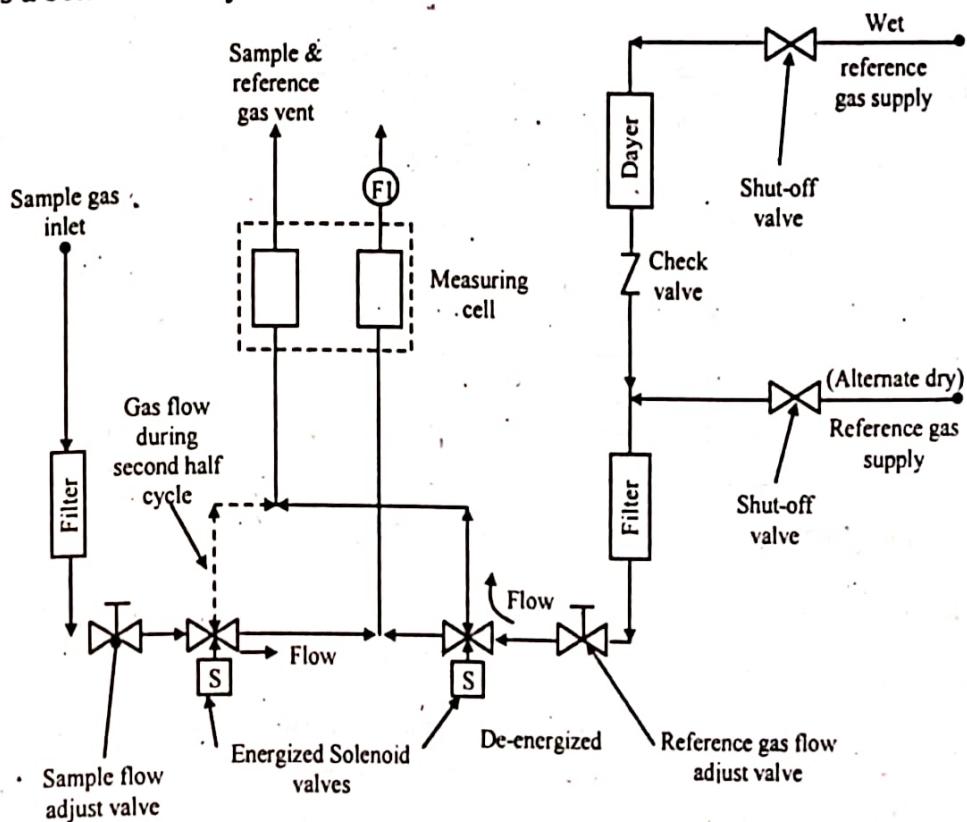


Fig: Piezoelectric hygrometer arrangement

b) Thermal conductivity method is quite conveniently used for the analysis of binary gas mixtures. In industry it is sometimes seen that a gas mixture contains more than two constituents all of which are varying in proportion quite independently. However, often the variation in the concentrations is interdependent and the problem then is equivalent to

that of a binary sample. Analysis by thermal conductivity method requires a reference gas. Air is usually chosen for the purpose and instead of actual conductivity values, relative values are indicated with air having a thermal conductivity reference value of unity. Analysis may be of single pass type where the binary mixture is passed through the sample only once and for successful analysis the thermal conductivity of the mixture must vary widely with the change in the composition and the thermal conductivities of the two constituent gases must be different. A relation for λ_m that is usually followed for this case is given by $\lambda_m = \lambda_1 \{1 + a(x_2/(1-x_2))\} + \lambda_2 \{1 + b((1-x_2)/x_2)\}$

where a and b are constants known as Wasiljewa constants and x_2 is the molar fraction of the second constituent. A few typical response characteristic curves are shown in fig below. Table below shows a few important binary mixtures and the reference comparison gases.

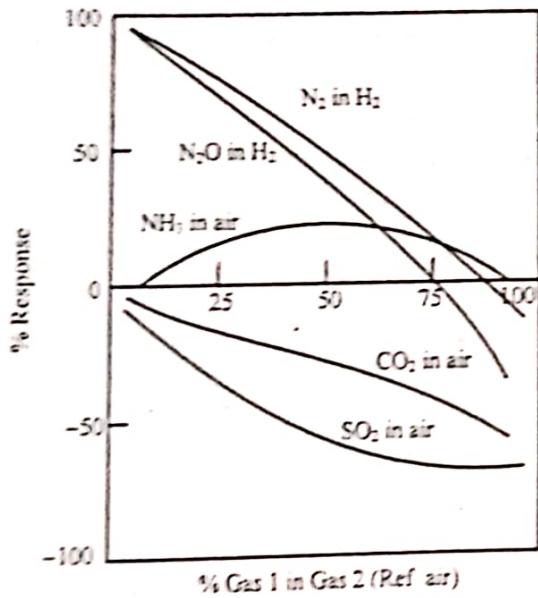


Fig: Response characteristics for binary mixtures

Table: Binary samples and comparison gases

Gas mixture	Comparison gas
1. H ₂ in air	H ₂ or air
2. H ₂ in N ₂	H ₂ , air or N ₂
3. H ₂ in CO ₂	H ₂ or CO ₂
4. H ₂ in Cl	H ₂ or Cl
5. H ₂ in methane	H ₂ , methane or a mixture of the two in appropriate proportion
6. He in air, O ₂ or N ₂	Any one of the constituent gases or H ₂ Air
7. Ne in air, Cl in air, acetone in air, NH ₃ in air, O ₂ in air	Air
8. SO ₂ in N ₂ or air	N ₂ or air
9. CO ₂ in flue gas, N ₂ or air	Air
10. Water vapour in air, O ₂ or N ₂	Air, O ₂ or N ₂

c) Measurement of density of solids, liquids and gases is very important in instrumentation. This is more so when they are continuously flowing from one site to another but it is very difficult to carry out the measurement of the density of solids under these conditions. Several techniques are available for the measurement of liquids and gases.

Mass density is a measurement, which can be defined as the mass per unit volume, whereas weight density is the weight-force per unit volume.

Specific gravity is known as the ratio of the density of the substance to the density of a water at 4°C. Specific volumes of all substances vary with temperature while those of compressible fluids are influenced by pressure also.

Density of fluid is the ratio of total mass to total volume. The measurements of densities of fluids are much more complex than solids. Therefore there are many different techniques developed such as Hydrometer, pycnometer, Hydrostatic weighing, Flotation methods, Drop methods and so on.

It is necessary to indicate the pressure and temperature at which the density is measured and wherever necessary, corrections are to be applied for obtaining the density at the standard temperature and pressure.

Apart from all conventional techniques available for the determination of the density of fluids in the laboratory, certain techniques suitable for use with some of the mechanical transducers or other electrical transducers. In this type of measurement variations in density is converted into either displacement, pressure or force, for further processing these for indication and other purposes.

Load cells can be used to weigh the container having a fixed volume of liquid when the container is provided with a continuous flow of liquid into it after suitably sampling from the main flow and an overflow device as shown in figure. Other techniques employ floats such as the hydrometer and other systems, resulting in output signals of pressure force or displacement.

Hydrometers are basically used for the measurement of specific gravity of a fluid i.e., the ratio of the density of the fluid to the density of the water.

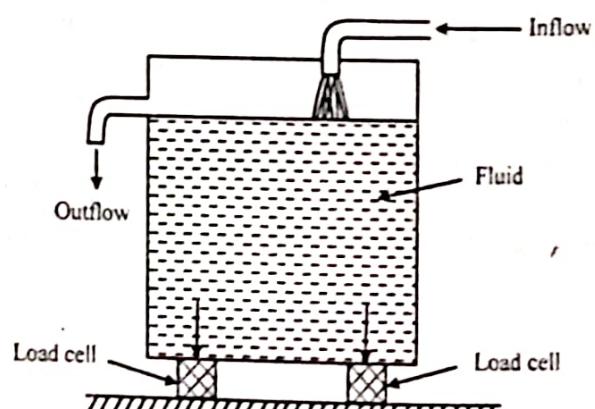
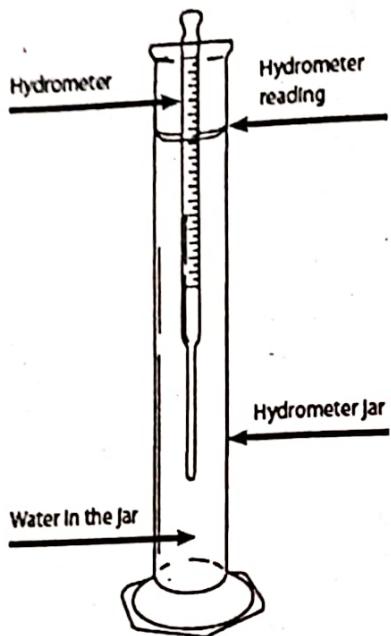


Fig: Density measurement by load cells



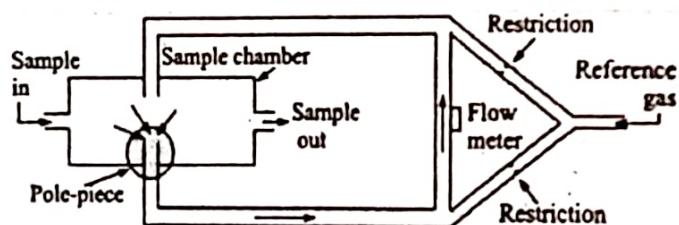
2. a) Explain with the help of a functional diagram, how can be oxygen concentration in sample gas determined by differential pressure type paramagnetic analyzer. Why does the instrument incorporate an electromagnetic with changing flux intensity?
 b) Draw & briefly discuss the different procedures of feeding the sample gas to the hot wire cell in case of hot wire TCD analyzer.
 c) Write some applications of hot wire TCD analyzer.
 d) Define kinematic viscosity & dew point.

[WBUT 2009]

Answer:

a) **Differential pressure type oxygen analyzer**

We know that the **oxygen gas** has its own **paramagnetic property**. The differential pressure type oxygen analyzer uses this property of oxygen. Here mainly two gasses are used, one is termed as **sample gas** and the other one is **reference gas**. Both gasses generate a pressure difference between them. The pressure difference gives rise to a flow in a horizontal tube and the flow rate, which is a function of the **percentage of oxygen** in the sample gas is measured.



(The above arrangement shows the Oxygen analyzer where the measurement is done on the basis of the pressure difference between two gases having different oxygen concentration in a magnetic field)

In the above arrangement air is used as the reference gas. Here one channel is kept under the strong magnetic field and the other one is completely free from any magnetic influence. Now, if both the sample and the reference gases are forced to flow through the sample chamber, on account of its paramagnetic property, oxygen gas builds up at the channel under the magnetic field causes a pressure difference between the two arms of the flow-tube. This leads to a flow in the channel connecting the two arms. This flow is measured using an appropriate transducer.

b) An arrangement dual hot-wire cell is shown in Fig. 1. It consists of two identical cells one fed from a standard sample gas reservoir, the other from the process column. The feeding of the gas to the cell can be done in a number of ways and the choice of a particular method is governed by the rapidly of response and accuracy. The design should be such that the measurement becomes independent of flow rate change and the response should be sufficiently fast. Three different techniques of sample-feeding are shown in Figs. 2 (a), (b) and (c). In Fig. 2(a), a convective exchange type cell is shown and it must be mounted such that the flow is vertical. In this case the measurement is usually independent of flow rate fluctuations. Figure 2 (b) shows a diffusion exchange type cell-system. The sensing and heating elements are separated from the main line by porous plugging and exchange takes place by diffusion through this plugging. This also is independent of flow rate fluctuation but the response time is considerably larger than that of Fig. 2(a). Figure 2(c) shows the direct online type. Here the response is rapid but the measurement is dependent on the main line flow rate.

The response equation of a hot wire cell is approximated by the relation

$$i^2 R = \alpha \lambda_m (T_s - T_a)$$

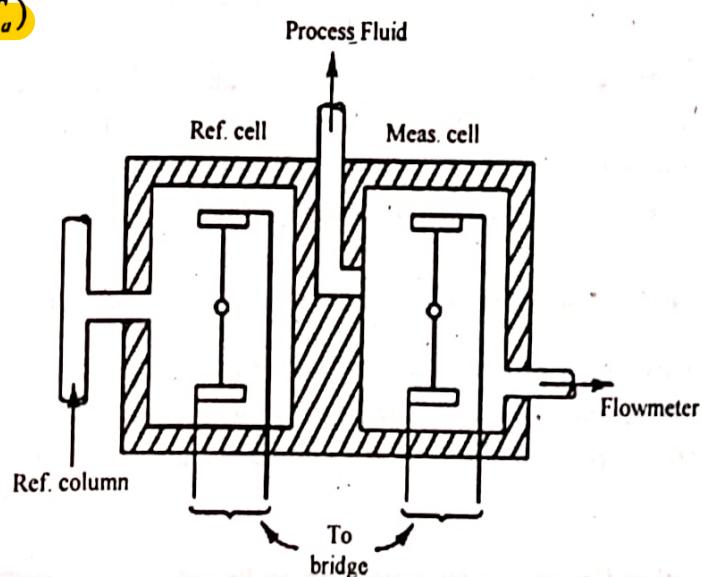


Fig: 1 A dual hot-wire thermal conductivity cell

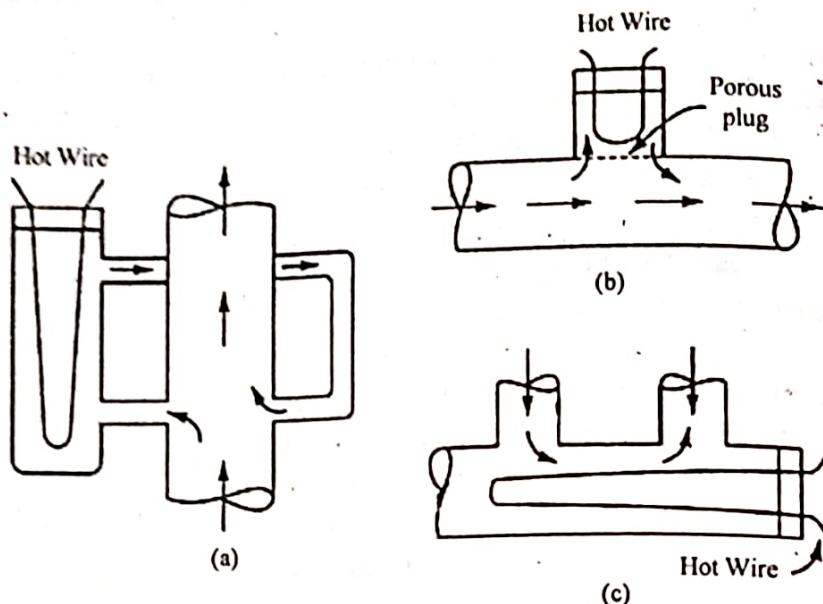


Fig: 2 Different techniques of feeding gas cell: (a) Convective exchange type, (b) Diffusion exchange type, (c) direct on-line type

In most of the instruments and other type of instruments the hot wire detectors are designed for the purpose of replacement science they are considered a consumable component.

The elements are often used in pairs or guards for insertion into a wheatstone bridge. In a two-hot-wire system, one detector senses the sampl gas, the second senses a reference gas. In a four hot wire system, two sensors are plumbed together (usually parallel) to the sample side, the other two sensors are similarly plumbed to the reference side,. To improve the analytic signal, the hot wire is usually presented as a coil.

c) The continuous thermal conductivity analyzer has wide application in industry where it is desirable to analyze a two gas mixture. The thermal conductivity method is very stable in regards to span and zero drift. Applications include welding shield gas mixtures, food packaging mixtures, leak detection mixtures and heat treating atmospheres.

d) Kinematic viscosity- The kinematic viscosity $\nu = \eta / \rho$ is defined as the ratio of the viscosity to the density of the fluid and is measured in units of m^2/s .

Dew point- The atmospheric temperature (varying according to the pressure and humidity) below which water droplets begin to condense and dew can form is called the dew point.

3. a) Define Newtonian liquid and Non-Newtonian liquid. [WBUT 2010, 2015]
Describe the operating principle of the Searle's rotating cylinder viscometer with necessary derivation. [WBUT 2010, 2012, 2015, 2016]

b) Draw the sketch of an electrolysis type hygrometer and explain its working principle. [WBUT 2010, 2015, 2016, 2018]

OR,

Write short note on Hygrometer.

[WBUT 2014]

c) How can concentration of the known component gas of binary mixture be found using thermal conductivity type gas analyzer?

[WBUT 2010]

Answer:

a) 1st part:

In continuum mechanics, a fluid is said to be Newtonian if the viscous stresses that arise from its flow, at every point, are proportional to the local strain rate — the rate of change of its deformation over time. That is equivalent to saying that those forces are proportional to the rates of change of the fluid's velocity vector as one moves away from the point in question in various directions.

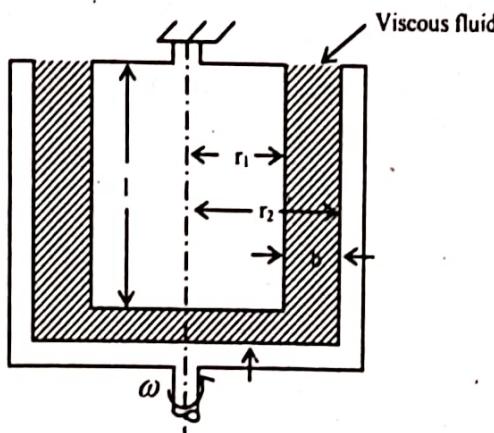
A non-Newtonian fluid is a fluid whose flow properties differ in any way from those of Newtonian fluids. Most commonly the viscosity (measure of a fluid's ability to resist gradual deformation by shear or tensile stresses) of non-Newtonian fluids is dependent on shear rate or shear rate history. However, there are some non-Newtonian fluids with shear-independent viscosity that nonetheless exhibit normal stress-differences or other non-Newtonian behaviour. Many salt solutions and molten polymers are non-Newtonian fluids, as are many commonly found substances such as ketchup, custard, toothpaste, starch suspensions, paint, blood, and shampoo.

2nd Part:

Searles rotating cylinder viscometer

It includes the measurement of torque produced by a cylinder rotated at constant speed in the viscous fluid. The torque may be converted into an electrical signal by connecting a motor driven shaft with cylinder through a spiral spring. Due to changes in viscosity, the motor shaft and the cylinder maintain an angular relationship, which is proportional to the torque on the spring.

For high viscosity fluids, the rotating cylinder method of Searle is used where the torque is measured by a mechanical method. This method is also known as **Couette concentric cylinder viscometer**. The sketch of such a method is shown in figure below.



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The inner cylinder is fixed while the outer cylinder rotates at constant velocity ωr_2 since the gap between the two cylinders is constant, the velocity gradient is also assumed uniform so that velocity gradient on the vertical and horizontal sides are $\omega r_2/b$ and $\omega r_1/b$ respectively, for both $b \ll r_1$ and $a \ll r_1$. The shear-stress-torque relation on the two sides are given by

$$\text{Horizontal} \rightarrow T_1 = S \cdot (2\pi r_1 l) \cdot r_1$$

$$\text{Vertical} \rightarrow T_2 = S \cdot (\pi r_1^2) \cdot \frac{r_1}{2}$$

The total torque is

$$T = T_1 + T_2 = \mu \omega r_1^2 \cdot \left[\frac{\pi r_1^2}{2a} + \frac{2\pi r_2 l}{b} \right]$$

By measuring T , angular velocity ω and apparatus dimensions r_1, r_2, l, a and b , the dynamic viscosity is easily evaluated. This type can measure viscosity from $10^{-3} - 10^{10}$ poise and having an accuracy of $\pm 1\%$.

Brookfield viscometer is a variation of this type where the inner cylinder rotates.

b) Hygrometer:

A typical electrolytic hygrometer shown below.

Principle: It utilizes a cell coated with a thin film of phosphorus pentoxide (P_2O_5), which absorbs water from the sample gas.

The cell has a bifilar winding of electrodes in a capillary tube. Direct current applied to the electrodes dissociates the water, which was absorbed by the P_2O_5 into hydrogen and oxygen. Two electrons are required for electrolyzing each water molecule and thus the current in the cell represents the number of molecules dissociated. A further calculation based on flow rate, temperature and current yields the parts-per-million concentration of water vapor.

The electrolytic hygrometer is desirable for dry gas measurements because it is one of the few methods that gives reliable performance for long periods of time in the low ppm and ppb regions. Electrolytic hygrometers require a relatively clean input gas, which usually necessitates the use of a suitable filter. The electrolytic cells should not be used with gases that react with P_2O_5 , such as NH_3 .

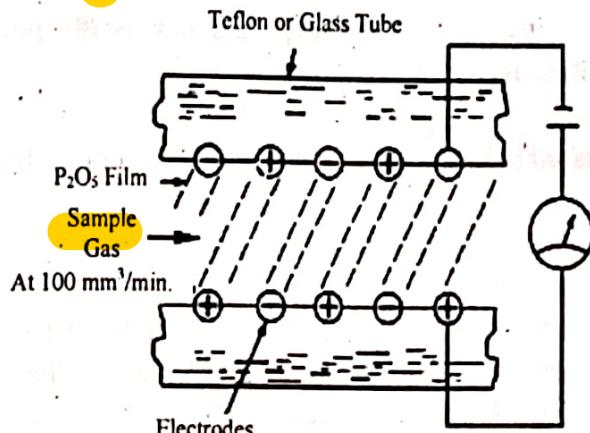


Fig. 1: Electrolytic hygrometer

The ability of the electrolytic hygrometer to measure water vapor content of a gas or gaseous mixture is based on the fact that a water molecule can be electrolyzed into molecular hydrogen and oxygen by the application of a voltage greater than 2V, the thermodynamic decomposition voltage of water. Since two electrons are required for the electrolysis of each water vapor molecule, the current in the electrolytic cell is proportional to the number of water molecules electrolyzed per second.

c) Refer to Question No. 1(b) of Long Answer Type Questions.

4. State the working principle of Zirconia oxygen analyzer with a neat sketch and also write the equation for the potential difference across the cell.

[WBUT 2010, 2018]

OR,

Describe the working principle of zirconia oxygen analyzer.

[WBUT 2014]

OR

Discuss the working principle of zirconia-oxygen analyzer with a neat diagram.

[WBUT 2016]

Answer:

Zirconia-cell O₂ analyzers are recommended for measuring photosynthetic O₂ evolution in rapid response system, because of their high sensitivity, stability.

Principle: It uses the principle that, when heated, zirconium oxide ceramic becomes a solid-state electrolyte through which O₂ molecules can migrate. The ceramic is formed into a hollow cup, with one side typically exposed to air and the other to the sample gas. As the O₂ molecules migrate across the ceramic wall of the cup, from the higher to the lower pO₂, they create an electric charge across the wall. The charge is picked up by a porous platinum film on either side of the cup. The resulting signal obeys the classic Nernst equation:

$$mV = (RT) \left[\ln(p_1 O_2 / p_2 O_2) \right] / (nF)$$

where R is 8.3144 J (mol·K)⁻¹, T is the temperature in Kelvin, n is the number of electrons dragged by each O₂ molecule (4), F is the Faraday constant, p₁ and p₂ are two

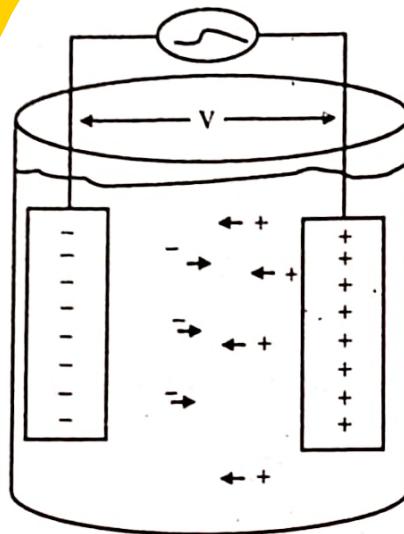


Fig: The movement of ions in solution under an applied potential. Anions move to the cathode and cations move to the anode

where resistance, R , equals the resistivity (ρ) multiplied by the length (l) between electrodes divided by the area (A) of that geometry between electrodes. The resistivity, also called specific resistance, has the units of ohms/centimeters. For solutions the area is usually taken as the area of each parallel electrode and the length becomes the distance between them. The conductance of a solution, G , is the inverse of the resistance, R , of the solution; therefore, $G=1/R$. As resistance is expressed in ohms (Ω), conductance is expressed in Ω^{-1} . The reciprocal ohm used to be called mho, but it is now officially called Siemens, S , and $1\text{ S} = 1\Omega^{-1}$.

The conductance of a sample is highly dependent on the cross-sectional area and the length between electrodes. The conductance will decrease as l increases and increases with cross-sectional area.

$$G = \frac{kA}{l} \quad \dots (2)$$

In the equation above, k is the conductivity. The units of k are S/m . This equation is also the inverse of the resistivity where $k=1/\rho=(1/R)(d/A)=G(d/A)=G\theta$. In this instance, θ , is equal to d/A in cm^{-1} . As θ is function of the geometry of the electrode and is a cell constant can be best determined by measuring solutions of known specific conductance ($k = G\theta$). Reference solutions for this purpose have been characterized in cells of known geometry.

As conductance is determined by the totality of ions present in a solution, largely acting independently of each other, it can be expressed as a summation. The k can be thought of as the conductance of one cubic centimeter of solution. Suppose 1 cm^3 contains 1 gram-equivalent of electrolyte. The equivalent conductivity Λ can be written in the following terms:

$$\Lambda = \frac{1000}{c} k$$

... (3)

The molar concentration is c and SI units of equivalent conductivity is Siemens meter-squared per mol (Sm^2/mol).

The cell constant can be defined as the ratio of the distance between the electrodes, d , to the electrode area, A . This however neglects the existence of a *fringe-field effect*, which affects the electrode area by the amount AR . Therefore $K = d/(A + AR)$. Because it is normally impossible to measure the fringe-field effect and the amount of AR to calculate the cell constant, K , the actual K of a specific cell is determined by a comparison measurement of a *standard solution* of known electrolytic conductivity.

The main advantage of AC excitation is that it can eliminate some of the error by using negative feedback to improve the stability.

In case of the AC amplifier, it can be made more stable at the signal levels delivered by sensors than DC amplifiers.

b) Molecular selective gas sensor to monitor dissolve carbon dioxide

The definition of a sensor can be given like - It is a device that is used to measures a physical quantity and converts it into an equivalent analog or digital signal which can be read by an observer or by an instrument.

In a **typical gas sensor**, it detects particular gas molecules and **produces an electrical signal whose magnitude is proportional to the connection of the gas**. But in practice there is no gas sensor exists that is **100% selective to only a single gas**. A good sensor is sensitive to the measurement quantity but less sensitive to other quantities. A variable gas sensor are based on five basic principles. These can be electrochemical, infrared, catalytic bead, photo ionization and solid-state.

A typical CO_2 sensor is given below:

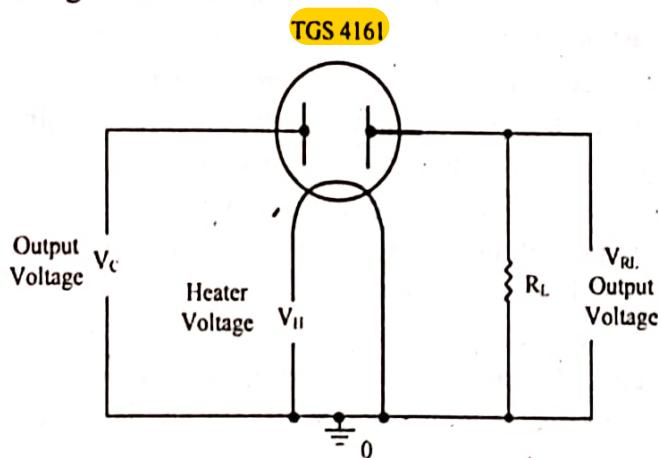


Fig: Circuit for CO and CO_2 sensor

CO₂ sensor

The operating circuit of CO₂ sensors is shown in fig. above. The relationship between output voltage and gas concentration is given by the following expression

$$C = \left[\left(\frac{(C_c R_L / V_{out}) - R_o}{R_o} \right) \frac{1}{K} \right]^2$$

Where, V_{out} = output voltage; C_c = input voltage, R_o = electrical resistance of sensors at zero ppm, K = a constant for particular, R_L = sensors load resistance.

6. a) Describe the operation of the thermal conductivity type gas analyzer. What is the function of the reference cell? [WBUT 2011, 2016]
b) Define specific gravity, kinematic viscosity and consistency. [WBUT 2011]
c) Explain the operation of the thermomagnetic method of oxygen analysis with necessary schematic diagram. [WBUT 2011, 2016]

Answer:

a) **Method of Measurement: Thermal conductivity measurement**

In the process of **thermal conductivity measurement**, the coefficients of thermal conductivity of the sample gas and a known gas mixture are compared. Owing to its experimental complexity and low accuracy, the absolute value of the coefficient of thermal conductivity is not measured.

The schematic diagram of a thermal conductivity analyser is shown in Fig. below. The sensing element (a resistor wire), situated at the middle of the cells. It is made of a material having a **high temperature coefficient of resistance** like platinum, tungsten, nickel or chromium coated copper.

The resistance wires are heated by sending a constant current through them. **The ratio of voltages of the two cells may be measured by standard methods.** Alternatively, the current terminals may be dispensed with and voltage terminals may be connected to the **two arms of a Wheatstone bridge.** The former method is preferred because it almost eliminates the so-called 'end cooling' of the resistance wires.

The percentage composition of **hydrogen and carbon dioxide in a mixture of two gases can be determined by** this. In this case, pure hydrogen is passed through both the cells at a certain flow rate and the ratio of voltages is set to 1:1. Next, the sample gas mixture is passed through the sample cell and hydrogen is passed through the reference cell while maintaining the previous flow rate for both.

Heat transfer from the hot wires to the metal block, maintained at a constant temperature, takes place by conduction through the gas and terminals ('end cooling'), convection, and radiation. Convection, owing its origin to gravity, can be minimized by using a cell of low diameter and keeping it horizontal. The radiation can be minimized by polishing the inner wall of the cell. In this way, in commercial thermal conductivity cells the various types of heat transfer are: convection – 2%, radiation – 1% and 'end cooling' – about

30%. But because of the comparison of thermal resistance changes of the reference and sample cells, these errors become negligibly small.

The bridge unbalance voltages can be calibrated for different gases at different concentrations. From these data the composition of an unknown mixture having known components can be figured out.

However, if a single cell is used to measure thermal conductivity then, assuming heat transfer takes place only by conduction through the gas, we can express the voltage on the measuring diagonal of an equal-arm Wheatstone bridge by

$$V = \frac{IRR_0R_0\alpha}{k(R+R_1)} \cdot \frac{\lambda_1 - \lambda_2}{\lambda_1\lambda_2}$$

where I is the current passing through the sensing element

R is the resistance of the sensing element in the cell

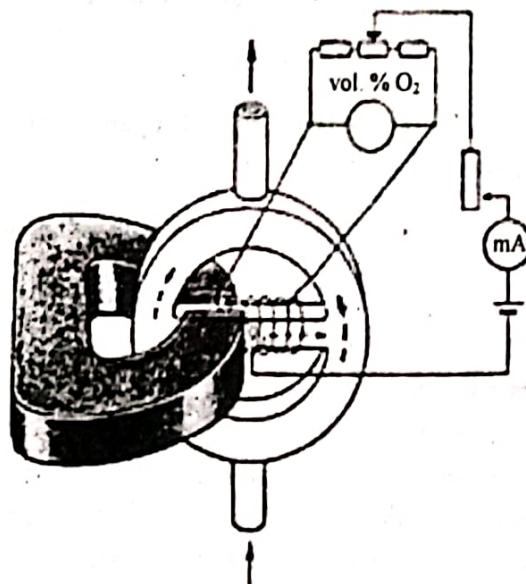
R_1 is the resistance of the contiguous arm of the bridge

R_0 is the resistance of the sensing element at 0°C

α is the temperature coefficient of resistance of the sensing element

k is the geometrical factor of the cell

λ_i is the thermal conductivity of the i -th component of the gas mixture.



b) Specific gravity: It can be defined as the ratio of the density of a substance to the density (mass of the same unit volume) of a reference substance.

Kinematic viscosity: The ratio of fluid's absolute viscosity to its density is known as the kinematic viscosity. The kinematic viscosity is the most common way of viscosity measurement.

It can be defined as:

$$\nu = \frac{\eta}{\rho}$$

Where

ν = kinematic viscosity

η = absolute viscosity

ρ = density in gram per cubic meter

SI unit of kinematic viscosity is meter-square-second

Consistency:

Quantitatively, consistency C_s is defined as

$$C_s = \frac{f}{w} \times 100 \quad \dots (1)$$

where f is the weight of the bone dry fibrous material present in a certain volume of the pulp/ stock slurry, and

w is the weight of the same volume of the pulp/stock slurry

It is important to be mentioned that a pulp slurry consists of fibrous material and water while a stock slurry contains additives, such as fillers and chemicals, over and above the pulp slurry.

c) Thermo magnetic Type Oxygen Analyzer

Magnetic wind-type oxygen analyser. Paramagnetic susceptibility χ_p of a substance depends on the absolute temperature T according to the Curie law

$$\chi_p = \frac{C}{T} \quad \dots (1)$$

where C is a constant. According to eqn. (1), the susceptibility of a paramagnetic substance decreases with increasing temperature.

This property of paramagnetic oxygen is utilized to generate a flow which has been termed as magnetic wind. Figure (i) shows the schematic diagram of such a magnetic wind generating oxygen analyser.

The instrument consists of a ring in the middle of which lies a horizontal glass tube. This horizontal tube houses two-wound resistors, which form two arms of a bridge. One of the wound resistors is placed between the pole pieces of a magnet and a current flows through the resistors so that they produce heat. In the absence of oxygen in a gas flowing through the ring, the wound resistors are heated to the same extent and the bridge to which they are connected remains balanced.

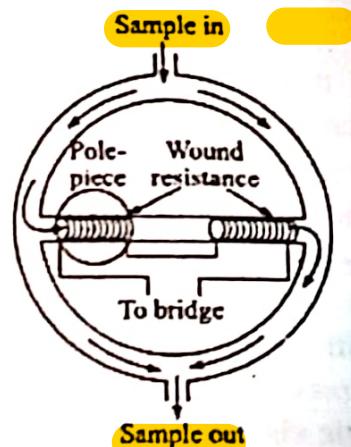


Fig: (i) Schematic diagram of magnetic wind-type oxygen analyser

If the sample gas contains oxygen, it is drawn to the left resistor where it gets heated to lose some susceptibility. Then the nearest cold gas on the left having more susceptibility gets drawn to the magnetic field, pushing the hot gas there to the right. Thus, a magnetic wind is generated from the left to the right of the horizontal tube. As a result, the left resistor region becomes colder than the right one, throwing the bridge out of balance. The resulting bridge unbalance current will be proportional to the percentage of oxygen in the sample gas.

The instrument, though robust, suffers from the following sources of error:

1. Heating and cooling of resistors are not only function of the flow rate of the magnetic wind, but also function of the composition and pressure of the sample gas. Different gases possess different thermal conductivities and viscosities which affect heat transfer and flow rate, upsetting the calibration.
2. Even if the background gases in the sample remain the same with variation in oxygen content only and pressure change is compensated for, the calibration curve becomes nonlinear at higher magnetic field strengths. Though it is linear at lower magnetic fields, but there the sensitivity is less (Fig. (ii)).
3. Hydrocarbon and other combustible gases in the sample react on the heating coils, degrade them and the calibration becomes questionable.

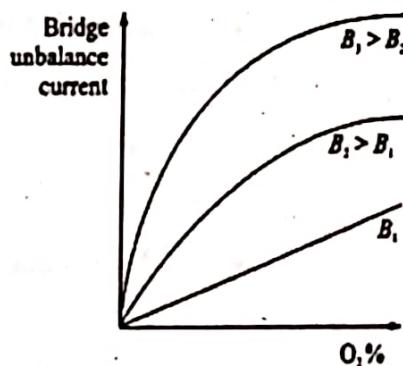


Fig: (ii) Bridge unbalance current vs. $O_2\%$ curves at different magnetic fields

This instrument is also called thermo-magnetic analyser.

7. How Zirconia cells as can be used oxygen analysis cells in power plant?

Make a comparative study of thermomagnetic method and zirconia cell method.

[WBUT 2012]

Answer:

1st part: Refer to Question No. 4 of Long Answer Type Questions.

2nd part:

Count	Thermomagnetic method	Zirconia method
1. Range	0 – 1% to 1 – 100%	0-1% to 0-100%
2. 90% response time	5 to 30 sec.	1 to 5 sec
3. Warm-up time	1 to 2 hrs	less than $\frac{1}{2}$ hr
4. Operating temperature range	0 to $50^\circ C$	1 to $1400^\circ C$
5. Scale	Almost linear	Logarithmic
6. Sampling requirement	yes	No
7. Operation affected due to Presence of gases like:	CO_2 for higher thermal conductivity	H_2, CH_4, CO - which are combustible

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8. Define Thermal conductivity of gas. How is this property used in the analysis of a gas? [WBUT 2012]

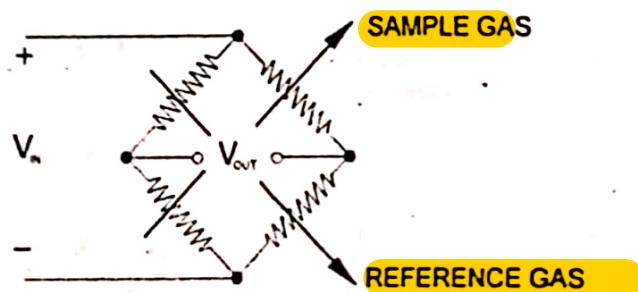
Answer:

1st part:

Thermal conductivity is the property of a material to conduct heat. Each gas has a known thermal conductivity - how well heat transfers through it.

2nd part:

The gas analyzer sensor uses four matched filaments that change resistance according to the thermal conductivity of the gas passing over it. These four filaments are connected in a Wheatstone Bridge configuration as shown below in Figure below



When all four resistances are the same, V_{out} is zero and the bridge is considered balanced. When zeroing, the reference gas is passed over all the filaments, the resistances will be the same (because filaments are matched) and the bridge is balanced. When the sample gas is passed over half of the bridge, depending on the thermal conductivity of the gas V_{out} will provide a non-zero output. Higher the thermal conductivity of the gas more heat from the filament will be carried out by the gas and more change in sensing resistance will occur causing more imbalance in bridge.

9. Write down some advantages and applications of thermal conductivity analyzer. [WBUT 2012]

Answer:

Advantages

1. Simple technology.
2. highly accurate thermal characterization of gasses
3. Non destructive

Applications

4. Measure the gas sample content of a sample mixture by comparing the thermal conductivity of the mixture with that of a reference.
5. To check the separation efficiency of the distillation tower.
6. Gas analyzer is used by industrial gas companies, metal heat treating companies and furnace manufacturers.

10. a) What is Dew point? Draw and explain about a commercial type dew point meter. [WBUT 2013]

OR,

Write short note on Dew point meter.

[WBUT 2014]

OR,

Explain the working principle of a dew point meter with schematic diagram.

[WBUT 2015, 2016]

b) Explain rotameter type viscometer with neat sketch. State the Poiseuille's formula. What is the main difference between viscosity and consistency?

[WBUT 2013]

c) Explain with neat sketch the Golay detector.

[WBUT 2013]

Answer:

a) 1st Part:

The atmospheric temperature (varying according to the pressure and humidity) below which water droplets begin to condense and dew can form is called the dew point.

2nd Part:

A method of dew point measurement is explained in Fig. 1. It measures the temperature of a polished surface when traces of condensation appear on its surface. The method is automated by a feedback system. It consists of a polished mirror in a chamber, which receives the gas whose dew point is to be measured. The gas is continuously supplied. There is provision for cooling or heating the mirror. Light falling on the mirror from a standard source is reflected onto a photocell. Another photocell receives light straight from the source. If the mirror is foggy due to condensation a difference signal is sent to the actuating unit to start the heater. If condensation has not started the freezer is on. A suitable temperature-measuring device records the temperature all the time. The system is automatic and always maintains the dew point very closely. The automation may be handled by microprocessor-based controller. It measures dew point in the range -40 to 25°C with an accuracy of $\pm 1^\circ \text{C}$.

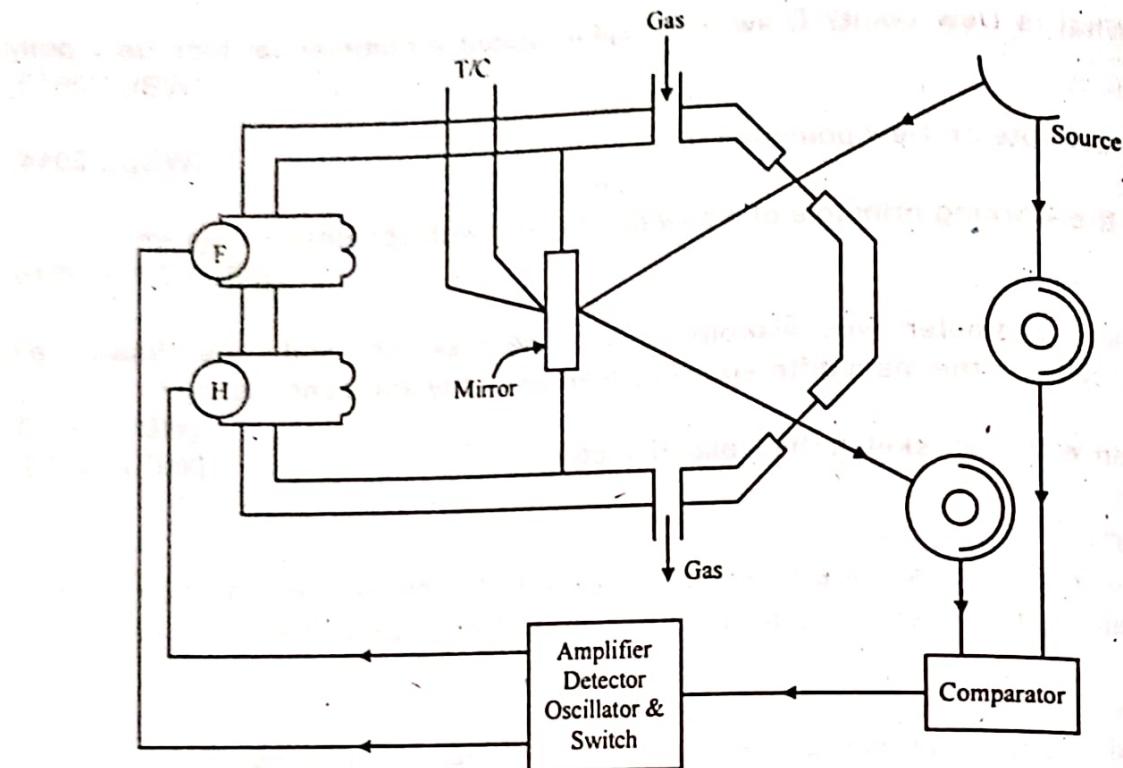


Fig: 1 Set up of a commercial type dew point meter

b) 1st Part:

Another continuous viscosity measuring system is the rotameter method. Rotameter bobs may be designed to be viscosity sensitive. Therefore with a rotameter in the line of flow, viscosity can be measured if flow rate is constant. Fig. 1 shows a scheme of the method. A rotameter with two bobs one sensitive and another immune to viscosity is mounted in a bypass line through which the flow is regulated by a suitable regulator. By adjusting the regulator, the flow rate in the bob is set at the index mark. The other bob will indicate the viscosity. If the range is required to be changed the flow rate can be changed and the scale reading can then be related to the viscosity by the empirical calibration. The calibration curves for three different flow rates is shown in Fig. 2. This method is commonly used up to a viscosity of 300 cp, but the range is extendable. It measures viscosity and also checks consistency at high static pressure.

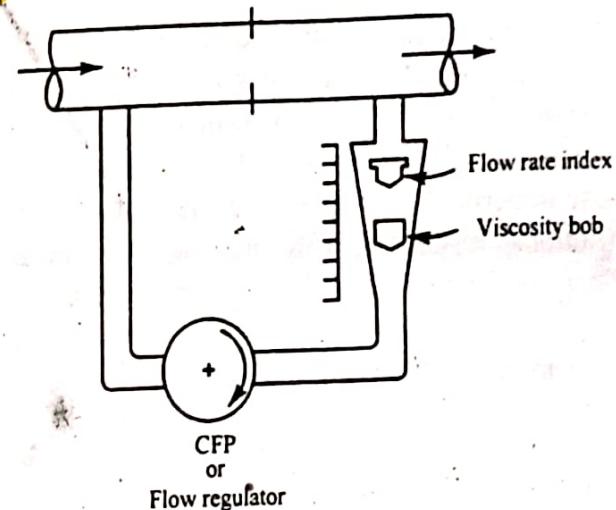


Fig: 1 Sketch of rotameter type viscometer,
CFP constant flow pump

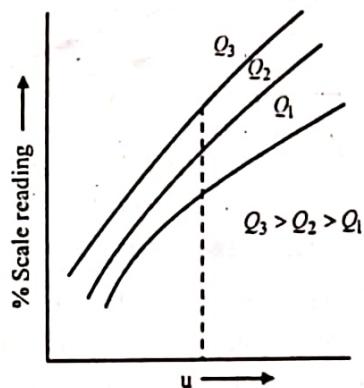


Fig: 2 Scale reading versus viscosity
curves for different flow rates

2nd Part:

Poiseuille's formula:

$$Q = \frac{\Delta P \pi r^4}{8\eta L}$$

where, ΔP = pressure difference

r = radius

η = viscosity

L = length

Note: The radius is by far the greatest influence on flow.

When the resistance increases, a greater pressure difference is needed in order to sustain flow at the same rate.

3rd Part:

Viscosity is a measure of the fluidity of the liquid or the gas. Many fluids undergo continuous deformation with the application of shearing stress. Application of a shear force, therefore, produces a flow. If this force-flow relation is linear, the fluid is Newtonian. For non-Newtonian fluids, the force flow relation is not only non-linear but changes from material to material. When continuous deformation occurs, the fluid tries to oppose this with a frictional resistance. This resistance can be measured in terms of consistency. Consistency of Newtonian fluids is called viscosity. Viscosity is typically different for different grades in a single process depending on the composition and is, therefore a convenient property to control product gradation.

c)

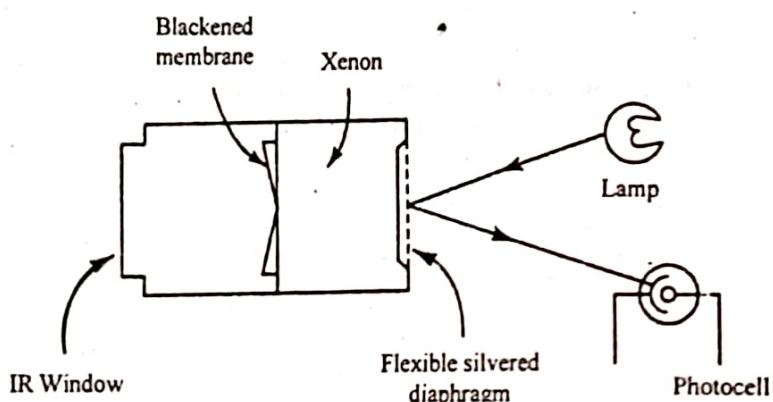


Fig: 1 The Golay detector

The principle of operation of Golay detector is shown in fig. 1. It is a gas thermometer that consists of cylindrical chamber filled with xenon and provided with a blackened membrane. One end of the cylinder has an IR window and the other a flexible diaphragm silvered on the outside. In the condition without IR radiation a beam of light is allowed to reflect from the silvered surface and to fall on the cathode of a phototube. With IR radiation entering the cell, the blackened membrane gets heated which is conducted into xenon gas increasing its pressure so that the silvered diaphragm gets distorted and consequently the fraction of light reaching the infrared beam strength. This detector is costly but is superior in performance in the range $\lambda > 50 \mu\text{m}$.

11. a) Explain the principle of operation of paramagnetic O₂ analyzer with a neat sketch.

b) Define Curie-Weiss law. In thermomagnetic O₂ analyzer, how old sample of O₂ is replaced by fresh sample of O₂? [WBUT 2013]

Answer:

a) A type of a paramagnetic gas analyser, often known as the magnetodynamic type, consists of a glass or quartz dumbbell suspended in a non-uniform magnetic field by a taut quartz fibre. The system is schematically shown in Fig. 1. The dumbbell is often filled with a diamagnetic gas like nitrogen.

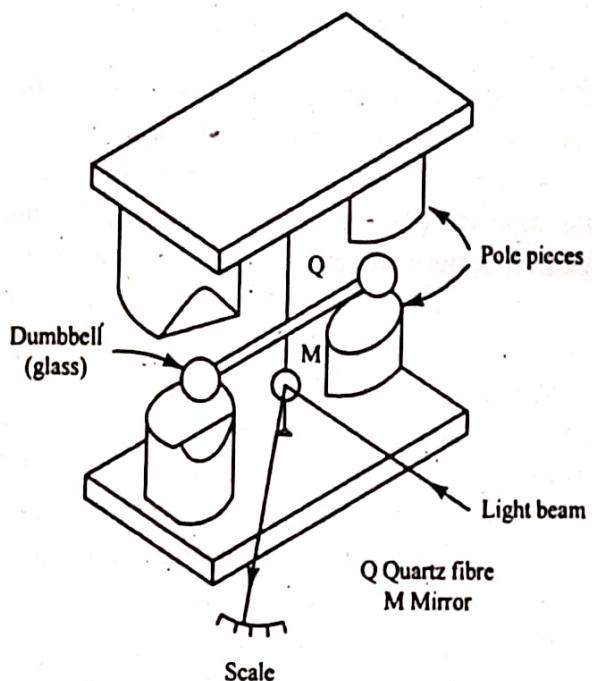


Fig: 1 Scheme of displacement type paramagnetic gas analyzer

The system is enclosed in a chamber through which the process gas is flushed at a specified slow rate. If the gas is not paramagnetic the force in the dumbbell is balanced. However, in presence of paramagnetic gases like oxygen, which will be attracted towards the pole pieces, the dumbbell will be displaced and a deflection of the suspended body occurs. The deflection of the body will be proportional to the percentage of the paramagnetic substance in the sample and this deflection is observed with a lamp-and-scale arrangement.

b) 1st part:

For paramagnetic substances, the susceptibility is independent of the strength of the magnetizing field but decreases with increase of temperature according to the Curie-Weiss law:

$$\text{Atomic susceptibility} = \frac{\text{relative atomic mass}}{\text{density}} \times \text{volume susceptibility}$$

$$= \frac{C}{(T - \theta)}$$

where T is the absolute temperature and C and θ are constants.

The susceptibilities of ferromagnetic materials vary with the strength of the applied field and above a certain temperature (called the Curie temperature and characteristic of the individual material) ferromagnetic lose their ability to retain a permanent magnetic field and show paramagnetic behaviour. The Curie temperature of iron is 1,000 K.

2nd Part:

The thermomagnetic oxygen analyzer is based on the principle that the magnetic susceptibility of oxygen decreases inversely with the **square of its temperature**. It consists of a test chamber containing two tubes for the entry of a test sample. The tubes are connected by a cross tube containing electrical heating filaments at each end of the crossover passage. The two filaments are the arms of a Wheatstone bridge. One end of the cross tube with its heating filament lies in a strong magnetic field created by the poles of a permanent magnet. The test sample is introduced in two equal streams through the two side tubes. Any oxygen in the sample is attracted to the magnetic field because the heating filaments, and the magnetic susceptibility of any oxygen in the sample decreased rapidly as the temperature is increased. The **heated sample is displaced by cool oxygen attracted to the magnetic field, and this flow of gas, or "magnetic wind," cools the filament in the magnetic field, causing its resistance to be different from the heating filament at the other (unmagnetized) end of the cross tube.** The difference in resistance is measured on a **bridge-type instrument**, and a signal proportional to the oxygen concentration in the test sample is transmitted to a recording or display instrument.

3rd Part: Refer to Question No. 6(c) of Long Answer Type Questions.

12. Explain with a diagram, the principle of operation of flame ionization detector and discuss the types of compounds best suited for this detector.

[WBUT 2013, 2015]

Answer:

The flame ionization (FID) detector is a detector used in **gas chromatography**. Such a detector is used to measure **hydrocarbon (HC) concentration** in the sample under study. The sample is brought to the detector via a pumping action with the help of a carrier gas through a hydrogen-air flame.

The FID passes the sample and carrier gas from **the chromatographic column**. The **hydrogen-air flame alone creates few ions**, but when a chemical compound is burned, it produces **ions and electrons**.

These are located between two **voltage-polarized electrodes** across which a potential difference of a few hundred volts, called as polarization voltage, is applied. As a result, the newly produced ions (**hydrocarbon**) generate a current that can be recorded. The intensity of the current is directly proportional to the amount of **analytes (ions)** present in the detector.

An **electrometer** senses this current, provides a voltage output using an operational amplifier, and sends to an output device.

Among other very extensively used detectors is the flame ionization detector where the eluate is combined with air and hydrogen to form a combustible mixture, which then is ignited to form a flame. This flame provides the energy to ionize the components in the eluted part from the column. The schematic of such a detector is shown in Fig 1. The cations are attracted towards the negative electrode kept around the tip of the flame called

the collector. The positive electrode is the metal burner or a metallic ring as shown. This flow of ions cause a current to flow in the external circuit which may be amplified and measured. The current is proportional to the number of ions collected by the negative electrode, which is proportional to the concentration of eluted component that is ionized. Thus it responds to organic samples where the number of C atoms are responsible for the positive ions produced. It does not respond to inorganic samples. In fact, their presence affects the performance of the detector. Carrier gas is inert. This detector is, as such, much more sensitive than thermal conductivity types.

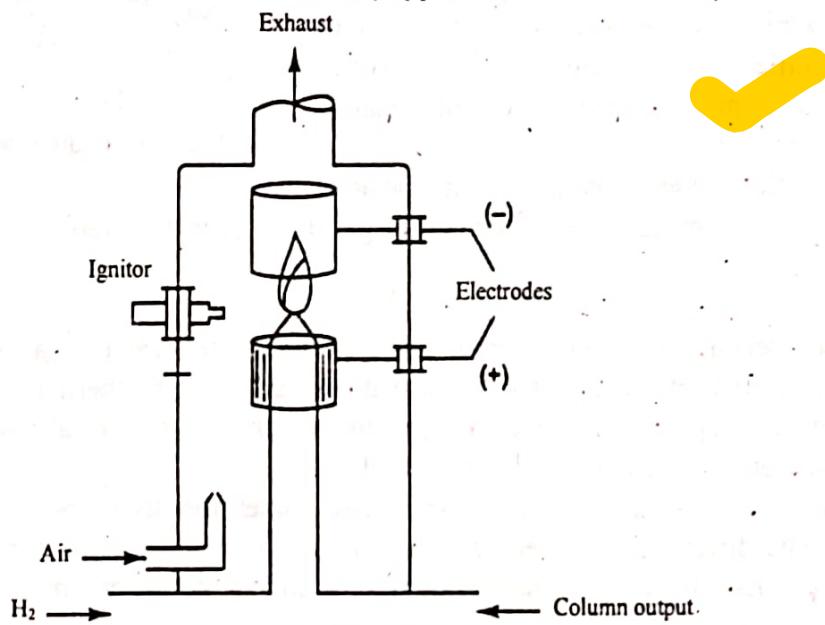


Fig: 1 The schematic diagram of a flame ionization detector

13. a) Briefly discuss the principle of thermal conductivity detector (TCD) and discuss the types of compounds best suited to be detected.
- b) Describe the mechanical force balance type oxygen analyzer with necessary diagram.
- c) Describe the working principle of Hydrogen Flame Ionization Detector (HFID) with necessary diagram. [WBUT 2014]

Answer:

- a) 1st Part:

Thermal conductivity detector (TCD)

The TCD is a truly universal detector. It consists of a heated sensor in a thermostated chamber, through which the effluent flows. Helium is usually used as a carrier gas, as it has the highest thermal conductivity of any gas, except for hydrogen. As the peaks elute, the thermal conductivity of the gas in the chamber changes. This changes the heat flow from the heated sensor, through the gas, to the walls. Since the sensor is being heated at a constant rate, it becomes hotter as the thermal conductivity of the effluent drops. The change in temperature of the sensing wire filament or thermistor changes its resistance. The sensor is wired into a Wheatstone bridge circuit and the change in resistance

produces an unbalance, which produces a signal. The circuit for the TCD detector is diagrammed in Figure.

The filament is sensitive to oxidation while heated and therefore must not have current flowing unless the carrier gas is passing through the chamber. The detector is limited by its relatively low sensitivity, compared to other detectors and usually has a fairly large dead volume. It is, therefore, not very suitable for capillary work. Because of these limitations the TCD is little used in environmental work, except for the determination of major constituents of air.

The sample flows over a heated wire in one arm of the Wheatstone bridge, while pure carrier gas flows over the reference wire.

2nd Part:

For proper performance, the thermal conductivity of the carrier gas should be very different from that of the analytes. Table 1 gives values of the thermal conductivities of some gases and vapours. Hydrogen and helium, which have a thermal conductivity much larger than that of most vapours, are preferred.

With gases like nitrogen or argon, which are sometimes used, poor results are often obtained. The differences between the thermal conductivities of the carrier gas and the analytes are small, the detector responses are low and the detection limits are also low.

Table 1

Thermal Conductivity of Gases and Vapours*

Gas or Vapour	λ
Hydrogen	7.1
Helium	5.53
Methane	1.45
Ammonia	1.04
Air	1
Nitrogen	0.996
Ethane	0.97
Acetylene	0.9
Propane	0.832
<i>n</i> -Butane	0.744
Methanol	0.727
<i>n</i> -Pentane	0.702
Carbon Dioxide	0.7
Ethanol	0.7
<i>n</i> -Hexane	0.662
Acetone	0.557
Chloromethane	0.53
Trichloromethane	0.328

* Relative to the Thermal Conductivity of air.

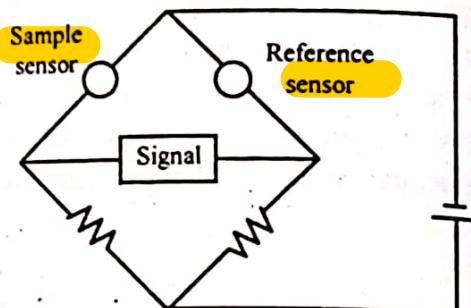


Fig: Thermal conductivity detector

b) **Magnetodynamic Oxygen Analyzer**

In the Pauling cell, two spheres of glass or quartz, filled with nitrogen, which is diamagnetic, are mounted at the ends of a bar to form a dumbbell. The dumbbell is mounted horizontally on a vertical torsion suspension and is placed between the specially shaped poles of a powerful permanent magnet. The gas to be measured surrounds the dumbbell. If oxygen is present, it is drawn into the field and so displaces the spheres of the dumbbell, which are repelled from the strongest parts of the field, so rotating the suspension until the torque produced is equal to the deflecting couple on the spheres (see Fig. 1). If the oxygen content of the gas in the cell changes, there will be a change in the force acting on the spheres, which will take up a new position. The magnitude of the force on the dumbbell may be measured in a number of ways, but a small mirror is commonly attached to the middle of the arm and the deflection is measured by focusing a beam of light on the mirror. The deflection may either be measured directly, or a force balance system may be used whereby the deflection of the dumbbell is detected but an opposing force is applied to restore it to the null position.

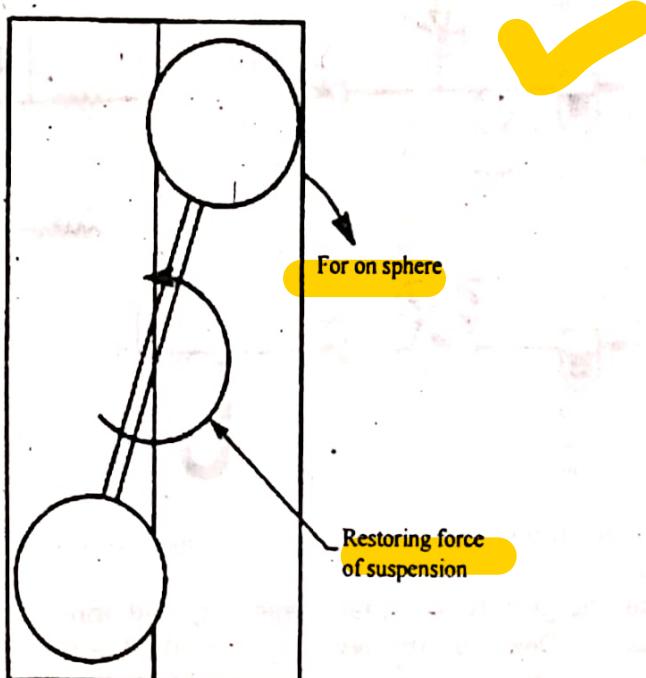


Fig: 1 Magnetodynamic oxygen measuring cell

c) Flame ionization detection (FID) is the most suitable means of analysis for most hydrocarbons at the levels found in polluted air. It may be used alone for the measurement of total hydrocarbons or as a detector after separation by a column device such as a detector after separation by a column device such as a gas chromatograph. In FID analyzers, a sensitive electrometer detects the increase in ion intensity in a hydrogen flame when air containing organic compounds is introduced. The response is approximately in proportion to the number of organically bound carbon atoms in the sample, so the detector is basically a carbon atom counter. Carbon atoms bound to

oxygen, nitrogen or halogens, however, give reduced response. There is no response to nitrogen, carbon monoxide, carbon dioxide or water vapor. The results are usually expressed in terms of the calibration gas used, e.g., parts per million (ppm) carbon as methane. Response is rapid and can be as sensitive as 0.1 ppm. The response to various hydrocarbons is not perfectly uniform and such variations should be taken into account in interpreting FID data. The FID analyzer is a reference method for total hydrocarbons in Air Quality Standards. Basically, it is an air metering device attached to a flame ionization detector. (figure below).

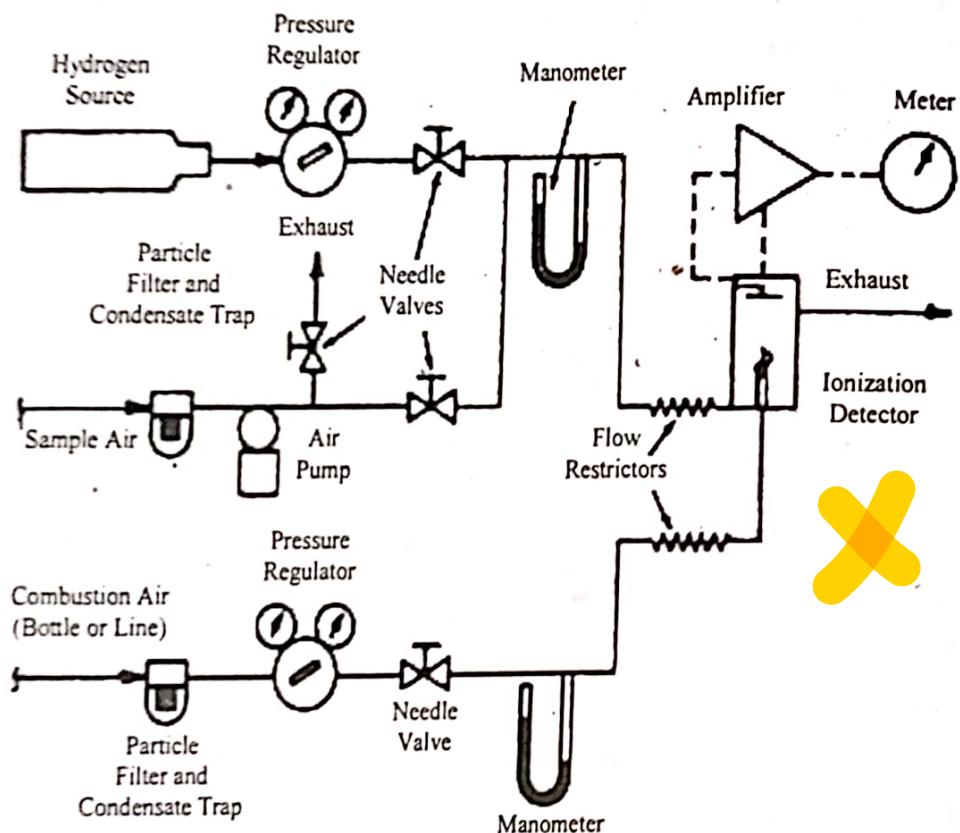


Fig: Hydrocarbon analyzer, hydrogen flame ionization detector

14. a) Define specific gravity, kinematic viscosity and consistency.
 b) Define dew point. Describe the working of Chilled-Mirror dew point hygrometer with necessary schematic diagram.

[WBUT 2014]

Answer:

a) Refer to Question No. 6(b) of Long Answer Type Questions.

b) 1st Part: Refer to Question No. 2(d) of Long Answer Type Questions.

Manual Chilled Mirror

Also known as the dew cup technique, this method uses a polished cup made of chromium plated copper. It is partially filled with acetone or methanol and a thermometer is placed in the solution. Small cubes of dry ice are dropped into the solution until

condensation forms on the outside of the cup. The dew point temperature is measured by reading the thermometer when the condensation begins to appear. This method is a one-time measurement and its accuracy is dependent upon the skill of the operator.

Optical Chilled Mirror Hygrometer

The optical chilled mirror device is capable of providing a continuous on-line humidity measurement over a prolonged period of time. Shown in figure below, it contains the following elements:

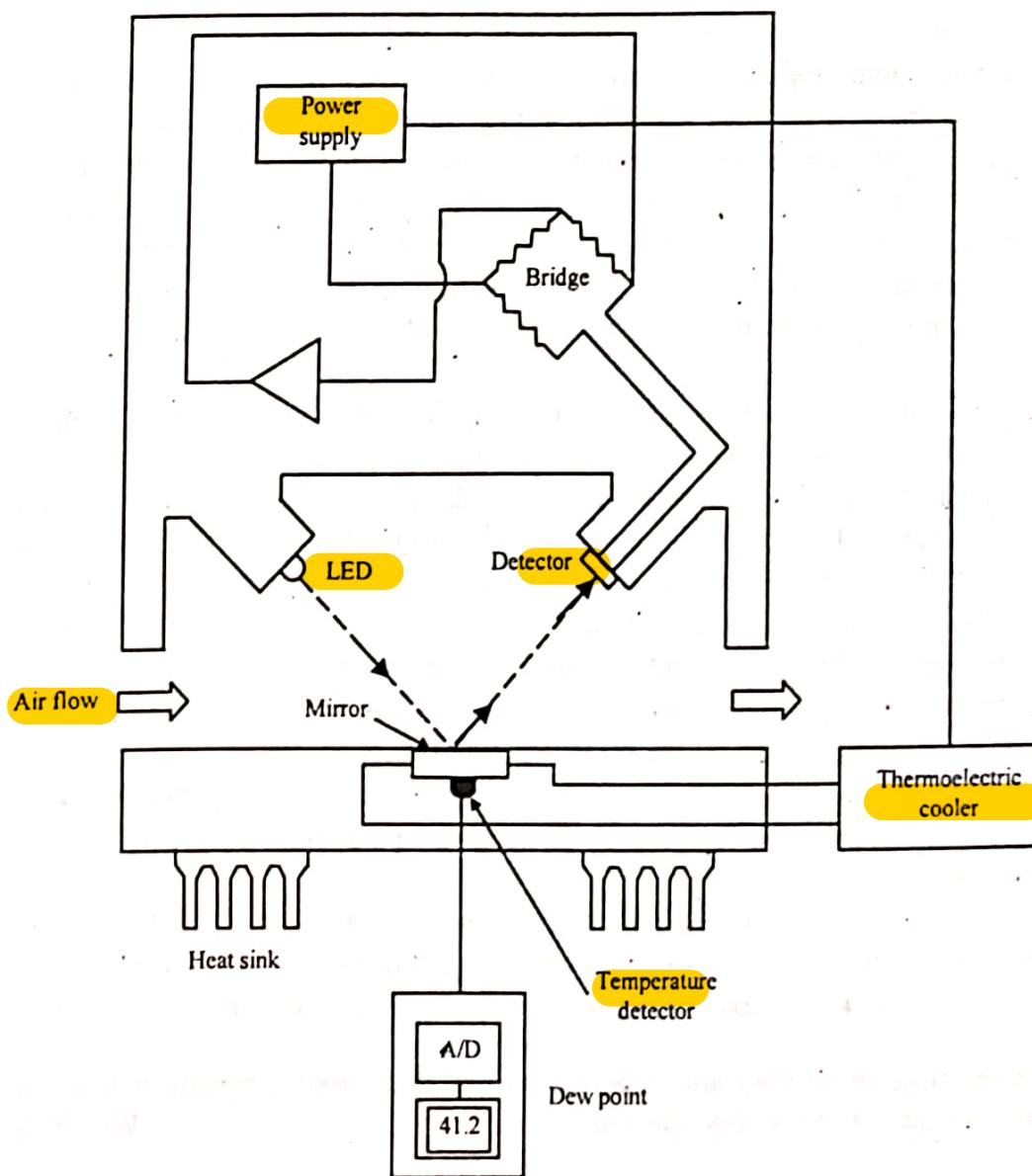


Fig: Optical chilled mirror hygrometer

Distillation

- Gold or rhodium-plated copper mirror
- A thermoelectric cooler used to control the temperature of the mirror

- A high intensity LED which shines its light on the mirror
- A phototransistor or an optical detector used to measure the amount of light reflected from the LED off the surface of the mirror
- The optical detector connected to one leg of an electronic bridge network that is coupled to an amplifier.

A flow of the sample air continuously passes over the surface of the mirror. When the mirror surface temperature is above the dew point, it is dry and highly reflective. Therefore, the maximum light is received by the optical detector. When the thermoelectric cooler reduces the mirror temperature, moisture will condense on its surface when the dew point is reached, causing the light to scatter due to refraction. Therefore, the light received by the detector is reduced. The change in the detector affects the bridge, which provides the feedback signal for closed-loop control. The purpose of the closed-loop system is to maintain the surface temperature on the mirror to within a few degrees of the dew point. This condition is performed by a four step cycling process.

Step 1: The mirror is rapidly cooled from above ambient to 1.5°C above the last dew point.

Step 2: The cooling rate is decelerated to approach and cross the dew point as slowly as possible to allow dew to form in a uniform manner.

Step 3: When the dew detection is completed, the current through the thermoelectric cooler is reversed. This action causes the mirror to rapidly rise in temperature until it reaches 1.5°C above the previous dew point level.

Step 4: The cooling cycle does not begin until the dew evaporates from the mirror surface and remains dry for a period of time. The mirror is dry for about 95 percent of the time, compared to the 5 percent time duration when the dew is present and the measurement is made. Typically, the measurement cycle is once every 20 seconds.

The temperature of the mirror's surface is measured by a platinum resistance thermometer embedded just beneath its surface. Its temperature is recorded and displayed at the moment the frost appears.

Because dew is on the mirror for only a short time, contamination buildup is kept to a minimum. Also, due to the cycling of the power, the life of the sensor is extended because it is not exposed to excessive heat for prolonged periods of time.

15. With the functional diagram, explain, in brief, the working principle of a hot wire thermal conductivity type gas analyzer. [WBUT 2015]

OR,

With the functional diagram, explain in brief, the working principle of a hot wire thermal conductivity type gas analyser. [WBUT 2017]

Answer:

The basic principle of operation of the system is the heat transfer from the heated wire to the cold surrounding fluid, heat transfer that is a function of the fluid velocity. Thus, a relationship between the fluid velocity and the electrical output is established. The

electronic circuit provides a controlled amount of electrical current to the wire and in the constant temperature method, to vary such a supply to maintain the wire temperature constant, when the amount of heat transfer varies.

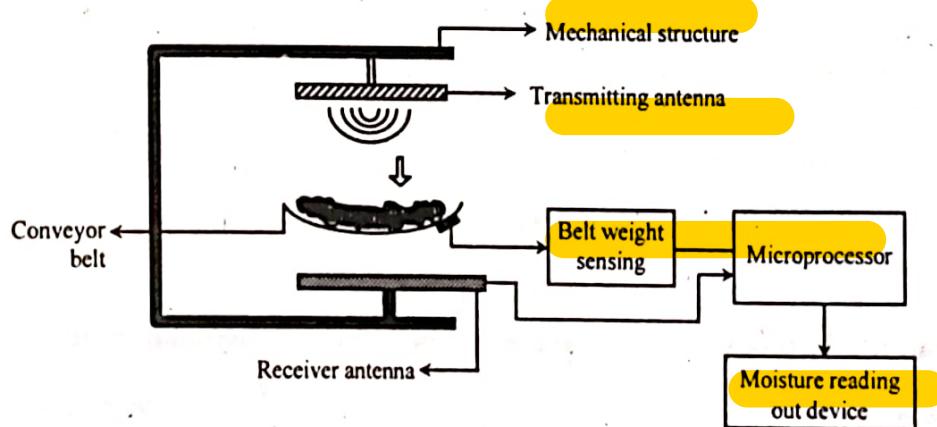
The TCD consists of an electrically heated filament in a temperature-controlled cell. Under normal conditions there is a stable heat flow from the filament to the detector body. When an analyte elutes and the thermal conductivity of the column effluent is reduced, the filament heats up and changes resistance. Wheatstone bridge circuit senses this resistance change and produces a measurable voltage change. The column effluent flows over one of the resistors while the reference flow is over a second resistor in the four-resistor circuit.

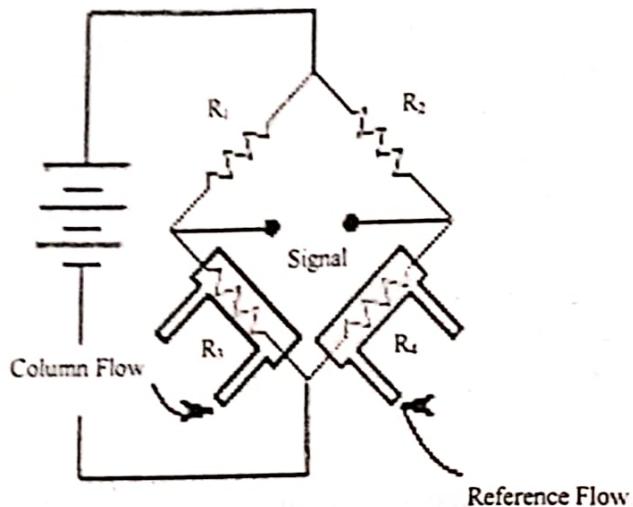
16. How can microwave be used for moisture measurement? Explain with neat sketch. [WBUT 2017]

Answer:

Microwave analyzers work on the principle that the water has a very higher dielectric constant compared to most other materials. When microwaves interact with water molecules within the material they slow down (and hence change phase) and weaken (attenuate) as the energy is transferred to the water.

An antenna transmits a beam through the material on the conveyor belt. A receiver antenna located below the conveyor belt receives the signal. The received signal is compared to the transmitted signal for phase and amplitude change and the microprocessor based control unit evaluates this signal. With the measurement of the above parameters and their evaluation, it is possible to determine moisture content and material density separately. The schematic arrangement is shown in the figure below.





17. a) The absolute viscosity of a fluid under test is 1000 centipoises. The density of the fluid is 0.8 gm/cm³. Calculate the following for the above fluid

- i) Fluidity in 'rhe'
- ii) Kinematic viscosity in 'stokes'
- iii) Relation viscosity in 'centipois'
- iv) Absolute viscosity in 'PAS'.

[WBUT 20]

Answer:

$$\text{Fluidity} = \frac{1}{\eta} = \frac{1}{1000} = 0.001 \text{ centipoise}^{-1}$$

$$\text{Kinetic viscosity} = v = 6.7197 \times 10^{-4} \mu/\gamma$$

where,

v = kinematic viscosity (ft²/s)

μ = absolute or dynamic viscosity (cP)

γ = Density (lb/ft³)

$$v = (6.7197 \times 10^{-4} \times 1000) / 0.8 = 0.75$$

1 centipoise [cP] = 0.001 Pascal second [Pas]

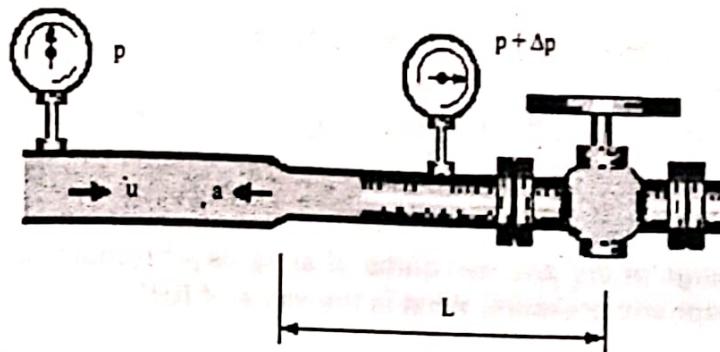
Therefore, 1000 cP = 1 Pas

b) Prove that in a capillary tube, the absolute viscosity of Newtonian fluid passing through it is given by $\mu = \frac{\pi R^4}{8VL} (P_1 - P_2)$.

[WBUT 20]

Answer:

A pressure surge in an elastic pipe will cause the pipe to swell and some of the energy will be absorbed by straining the pipe wall. This reduces the rise in pressure. The more elastic the wall is, the less the pressure rise will be. Consider the case shown.



$$\text{Kinetic Energy lost by fluid} = \frac{1}{2} \rho u^2$$

$$\text{The mass of fluid is } \rho A L \text{ so substituting K.E.} = \frac{1}{2} \rho A L u^2$$

$$\text{Strain Energy of fluid} = \Delta p^2 A L / 2K$$

Now consider the strain energy of pipe wall. The strain energy of an elastic material with a direct stress σ is given by

$$SE = (\sigma^2 / 2E) \times \text{volume of material}$$

The pipe may be regarded as a thin cylinder and suitable references will show that stress stretching it around the circumference is given by the following formula.

$$\sigma = \Delta p D / 2t$$

$$\text{Volume of metal} = \pi D t L$$

$$\text{Hence, S.E.} = \left(\frac{\Delta p D}{2t} \right) \times \frac{\pi D t L}{2E} = \frac{(\Delta p)^2 D A L}{2t E}$$

Equating KE lost to the total S.E. gained yields

$$\frac{\rho A L u^2}{2} = \frac{(\Delta p)^2 D A L}{2t E} + \frac{(\Delta p)^2 A L}{2K}$$

$$\rho u^2 = \frac{(\Delta p)^2 D}{t E} + \frac{(\Delta p)^2}{K} = (\Delta p)^2 \left\{ \frac{D}{t E} + \frac{1}{K} \right\}$$

$$\Delta p = u \sqrt{\left\{ \frac{\rho}{\frac{D}{t E} + \frac{1}{K}} \right\}}$$



The solution is usually given in terms of the effective bulk modulus K' which is defined as follows

$$K' = \left\{ \frac{D}{t E} + \frac{1}{K} \right\}^{-1}$$

The pressure rise is then given by $\Delta p = u[\rho/K']^{\frac{1}{2}}$

The acoustic velocity in an elastic pipe becomes a' and is given as $a' = (K'/\rho)^{\frac{1}{2}}$

Hence $\Delta p = \rho u a'$

18. a) The readings of dry and wet bulbs of sling psychrometer are 18°C and 16°C at normal atmospheric pressure. What is the value of RH? [WBUT 2017]

Answer:

The table Relative Humidity - Temperature in Celsius below is used to estimate the relative humidity of air if dry and wet bulb temperatures are known.

$T_{db} - T_{wb}$ (°C)	Relative Humidity - RH(%)							
	Dry Bulb Temperature - T_{db} (°C)							
15	18	20	22	15	27	30	33	
1	90	91	91	92	92	93	93	
2	80	82	83	84	85	85	86	87
3	71	73	75	76	77	78	79	80
4	62	65	67	68	70	71	73	74
5	53	57	59	61	64	65	67	69
6	44	49	52	54	57	59	61	63
7	36	42	45	47	51	53	55	58
8	28	34	38	41	45	47	50	53
9	21	27	31	34	39	41	45	48
10	13	20	25	28	33	36	40	43

According to the data given in the question, dry bulb temperature (horizontal line) is 18°C and difference between dry bulb temperature (T_{db}) and wet bulb temperature (T_w) (left side Vertical line) i.e. $(T_{db} - T_{wb}) = (18 - 16)^\circ\text{C} = 2^\circ\text{C}$

The table says that the RH = 82%

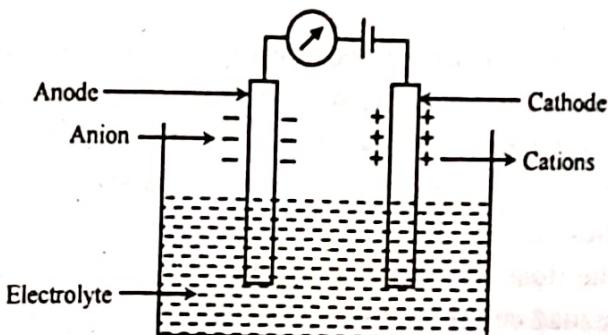
b) Define Newtonian fluid and non-Newtonian fluid. Describe the operating principle of the Searle's rotating cylinder viscometer with necessary derivation. [WBUT 2017]

Answer: Refer to Question No. 3(a) of Long Answer Type Questions.

c) Prove that the relation between water vapour content in air and the electrolytic current is linear in case of electrolytic hygrometer. [WBUT 2017]

Answer:

Electrolytic hygrometer is based on Faraday's Laws of Electrolysis that says the passage of electricity through an electrolyte with cations moving to the cathode to get reduced and anions moving towards the anode to get oxidized where electrolyte is the liquid that conducts electricity.



In other words, the amount (m) of a substance deposited, evolved or dissolved at an electrode is directly proportional of electricity (Q) passed through the electrolyte.

$$m \propto Q$$

It is known that $I =$ current through the electrolyte (Electrolytic current)

$$= \frac{Q}{t}$$

$$\therefore m \propto It$$

$$\text{or, } m = zIt$$

where, $z \Rightarrow$ Electrochemical equivalent of the substance

According to Faraday's law, when the same quantity of electricity is passed through several electrolysis, the mass of the substance deposited an proportional to their respective chemical equivalent.

In **electrolytic hygrometer**, moisture containing air is passed around the platinum electrodes with phosphorous pentoxide (P_2O_5) that absorbs water when voltage is applied to the electrodes of water decomposes into hydrogen and oxygen. Thus, according to Faraday, Electrolytic current is proportional to the amount of water.

$$\therefore m \propto I$$

19. Write short notes on the following:

- a) Hydrometer [WBUT 2009]
- b) Katharometer [WBUT 2009, 2013, 2014, 2016, 2017, 2018]
- c) Psychrometer [WBUT 2010, 2018]
- d) Toroidal method of conductivity measurement [WBUT 2012]
- e) Vibrating U tube densitometer [WBUT 2012, 2016]
- f) Ostwald viscometer [WBUT 2012]
- g) Zirconia Cell Oxygen Probe [WBUT 2015]
- h) Flame ionisation detector [WBUT 2017]
- i) NDIR [WBUT 2018]

Answer:

a) Hydrometer System:

The diagram below shows the arrangement of a hydrometer which consists of a glass float that is weighted at the bottom with mercury or lead balls to make it float upright, as shown in Figure 1(a). The float has a hollow stem inside – a graduated scale – which serves to indicate the density of the liquid. If the level of the liquid in the container is

held constant by using an overflow tube, the vertical displacements of the float end stem can be used for indication of the density. A short steel needle attached to the stem will serve as the moving iron needle of the linear variable differential transformer that translates the displacements into an electrical signal. The system is calibrated by using liquids of known densities.

The bayonet force on the float acting along the axis of the hydrometer stem can be used to load a cantilever type load cell if the force is of reasonable magnitude.

Another arrangement that consists, three floats, each differing in its volume and density from the others, are connected together to form a single assembly while having the freedom to rotate, as shown in Figure 1(b). The bayonet forces working on all the three decide the angular position of the assembly and by suitably designing the sizes of the floats, it is possible to make the angular deflection proportional to the density of the liquid.

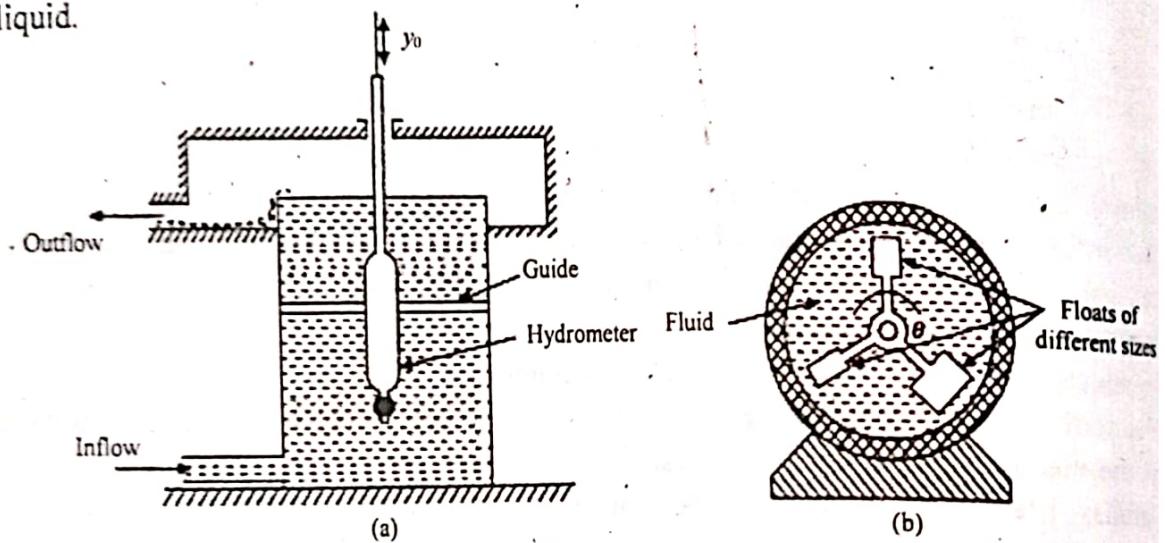


Fig: 1(a) Hydrometer system for density measurement;
(b) Three-float hydrometer system

b) The Katharometer or Thermal Conductivity Detector:

The thermal conductivity detector is a simple and most widely used type of detector. The principle behind the Thermal conductivity detector is- all gases have the ability conduct heat, but in varying degrees, this difference in heat conduction can be used quantitatively determine the composition of a mixture of gases. By definition, the thermal conductivity of a gas is the quantity of heat (in calories) transferred in unit time (second) in a gas between two surfaces 1 cm^2 is area, and 1 cm apart, when the temperature difference between the surfaces is 1°C .

A typical arrangement of the detector is given below. It consists of a hollow tube with electrically heated coil mounted axially in its centre. When only the carrier gas flows over it, a thermal balance can be attained at a certain temperature. When a gas or vapor differing in thermal conductivity from the carrier gas flows past the heated coil, the temperature of the coil gets altered and a proportionate change in the electrical resistance of the wire takes place. Such changes in resistance arising from the components of the sample are used for detection and estimation of the unknown sample components.

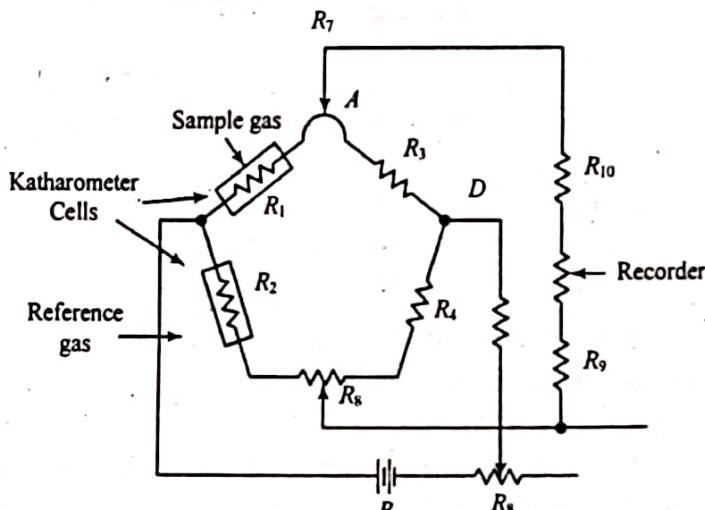
In practical case, the detector consists of two temperature sensing elements arranged in a Wheatstone Bridge circuit, one in the reference and the other in the measuring arm. The heat-sensitive elements are either thermistors or resistance wires, like platinum or tungsten. Figure below shows the detail circuit arrangement for measuring the changes in the resistance produced in the Katharometer cell elements. Resistances R_1 and R_2 are the Katharometer wires, resistances R_3 and R_4 are the ratio arms of the bridge used for making the base line adjustment and are made of manganin wire. The output of the bridge is fed to the recorder through an attenuator, so that if signal is greater than the span rating of the recorder, full-scale reading may be adjusted.

For the balanced bridge conditions, when the carrier gas flows through the two cells, no current would be flowing between A and C and:

$$R_1 / R_2 = R_3 / R_4$$

When the resistance R_1 changes due to the components of the sample gas, it causes an unbalance current to flow from A to C . The magnitude of the current serves to detect and measure the magnitude of the gas component vapour passing over the measuring cell. If the Wheatstone bridge is excited with an ac current, it can be made many times more sensitive, because the ac signal can be conveniently amplified before it is given to the recorder. The sensitivity of a thermal conductivity detector depends upon the nature of the carrier gas. When helium gas is used,

10^{-7} g of inorganic gases can be detected.



c) Psychrometer:

Psychrometer is a humidity measurement method, mainly measures relative humidity. It has a classical and a commercial methods of measurement.

A commercial type of psychrometer is shown in Fig. below. The filled system thermometer bulbs are placed adjacent to each other and an air draft is given through a blower, or they are placed where there is a strong draft, about 300 m/min. One of the bulbs is coated with knitted or woven cotton wick and is suspended into water reservoir whose level is controlled such that the requisite conditions mentioned earlier are satisfied. The temperatures are recorded by a suitable recorder.

The wicks accumulate dirt or/and other dissolved materials easily and get stiffened to lose water absorption capacity. The stiffened wicks can be reconditioned by boiling in hot

water. Instead of wick, the bulb can be soaked by enclosing it in a porcelain sleeve and water is pumped into the annular space at a constant rate.

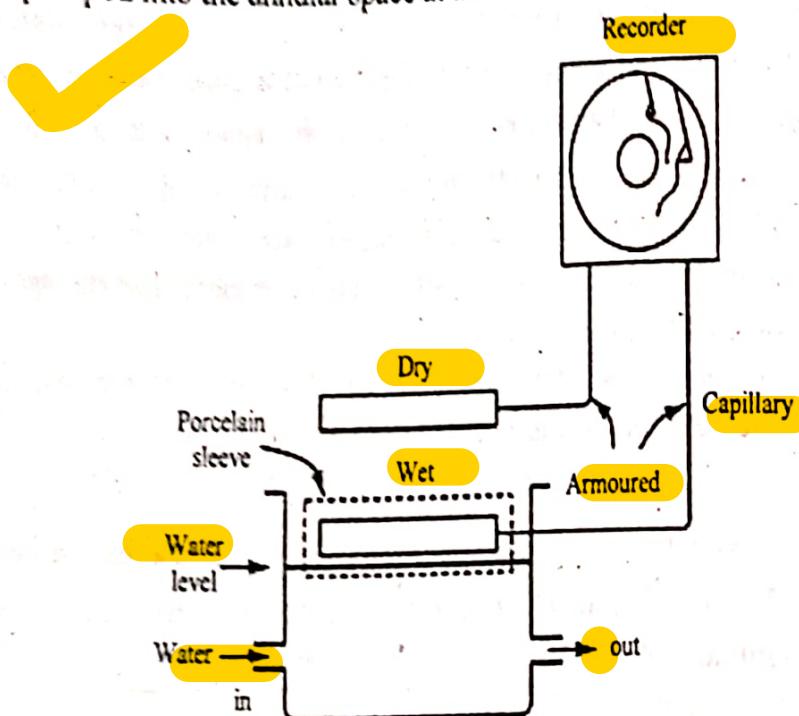


Fig: Sketch of a commercial dry and wet bulb psychrometer

On the other hand, in case of the classical method, the instrument consists of two bulb thermometers. One thermometer bulb is kept dry and is allowed to read the room temperature. The other bulb is kept wet by a water soaked wick and this thermometer measures temperature of adiabatic saturation, i.e., the temperature when thermodynamic equilibrium is reached between cooling by evaporation and heating by convection. The wet thermometer is given a large draft of air for this purpose so that the floating air will not tend to cling to the moist surface. The calculation of H_R from the difference of temperatures is quite complicated and is approximately obtained from the equation

$$P_v = P_{sat} - kP_T(t_d - t_w) \quad \dots (1)$$

where P_v is the partial vapour pressure; P_{sat} is the saturation partial pressure and P_T the total pressure, all expressed in mercury height; the term t_d and t_w dry and wet bulb temperatures respectively in °F; and k is a constant. Unfortunately k is also dependent on t_w . In fact k is of the form

$$k = A[1 + B(t_w - 32)] \quad \dots (2)$$

where A and B are two empirical constants. Values of P_{sat} and P_T are generally found in psychrometric table and then using Equation (1) and (2), P_v is found out, and finally

$$\%H_R = (P_v / P_{sat}) \times 100 \quad \dots (3)$$

is obtained. However, psychrometric charts are also available which give directly the approximate H_R -values for the reading of t_d and t_w .

thermometers are fastened to a light base which is suitably stringed to a handle such that about this handle the base can be whirled for generating the required draft of air. This type of psychrometer is used in air-conditioning systems for maintaining humidity at a specific value.

d) **Toroidal method of conductivity measurement:**

In a toroidal conductivity measurement cell, the electrodes are located outside the solution being measured. An oscillating potential is applied to the first electrode, that induces a current in the solution, on the other hand the induced current is measured with the second toroidal coil.

In food industries this types of instruments are used largely.

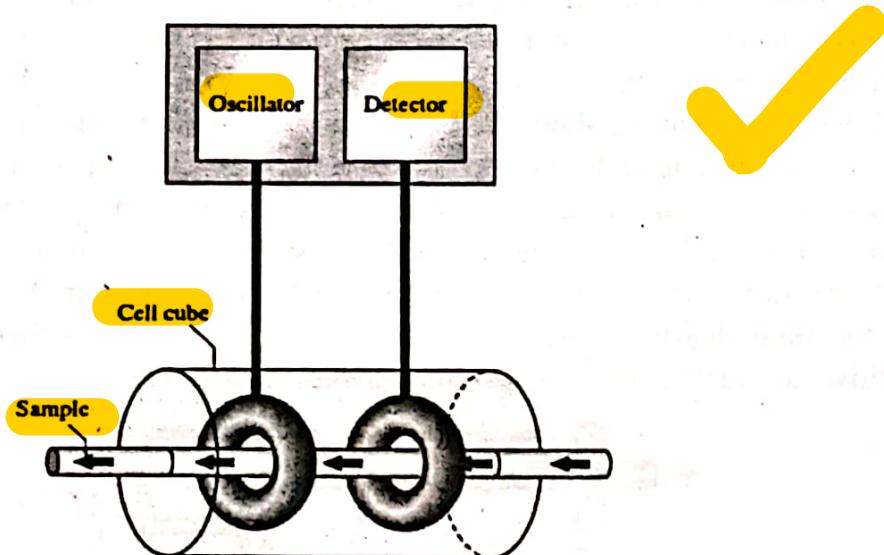


Fig: Torodial inductive conductivity cell

Characteristics of Toroidal Conductivity. The major advantages of toroidal conductivity is that the toroidal coils are not in contact with the solution. They are either encased in a polymeric material or external to the solution, as is the case with a flow through sensor. The toroidal sensor can be completely coated by a solid or oily contaminant in the process, with essentially no decrease in the reading until the coating builds up to a thickness of 1 cm. The polymeric material housing the toroids, or the pipe material in a flow through configuration, can be chosen to be resistant to corrosive solutions, which would quickly corrode contacting sensors with metal electrodes.

The major drawback to the toroidal conductivity measurement has been its lower sensitivity compared with contacting conductivity, and, although some recently developed toroidal conductivity analyzers can measure low conductivity, their application has been restricted to those rate applications where the process has low conductivity and is fouling.

Toroidal sensors are also typically larger than contacting sensors and the solution current induced by the toroid occupies a volume around the sensor. So toroidal sensors need to be mounted in a larger pipe than a contacting sensor. If the toroidal sensor is mounted less than a sensor diameter from a conducting pipe, there can be an upscale reading from conduction through the pipe. Near a nonconductive (plastic) pipe, the readings can be low because of reduction in the conductive solution volume near the sensor. Both of these effects can usually be corrected for, by zeroing the measurement loop with the sensor mounted in the dry process pipe.

e) **Vibrating U tube densitometer:**

The arrangement below shows the process of density measurement with vibrating U tube. A different, low cost/low accuracy concept is used in the detector here. A $\frac{1}{2}$ in. (12.5 mm) diameter U-tube section is present here which is welded at the node points, through which the process fluid flows continuously. The total mass of the U-tube assembly is affected by the process fluid density. A pulsating current through the drive coil brings the U-tube into mechanical vibration. An increase in process density increases the effective mass of the U-tube and, thus, decreases the corresponding vibration amplitude. An armature and coil arrangement is provided to detect the vibration at the "pickup" end. The armature vibrates together with the U-tube and induces an AC voltage proportional to the fluid density in the pickup coil. This AC voltage is then converted into DC millivolts, which is more compatible with remote recorders or controllers.

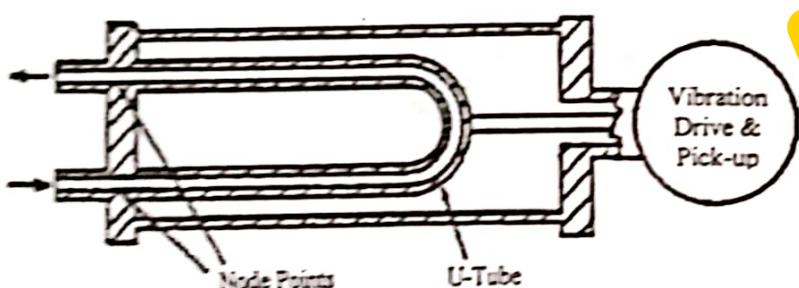


Fig: Vibrating U-tube density detector

In installations where the process temperature is expected to vary, an automatic temperature compensating circuit performs the required temperature correction. If the process stream contains entrained gases, then low flow velocities can cause separation or trapping of the gas. The measured value then represents some average value of the liquid and gas density. As with most vibrating densitometer designs, the entrained gas influence on the measured density can be compensated to some degree by offsetting the measurement result, provided that line pressure and flow remain suitably constant. Several different materials for the wetted parts are available for this densitometer, e.g., for applications with aggressive liquids or abrasive slurries. This densitometer can handle homogenous fluids or light slurries with low or moderate viscosities. High-viscosity streams or heavy slurries are likely to plug the small diameter U-tube. If the densitometer

is used in a bypass configuration, proper means have to be provided to secure a representative sample flow through the bypass.

f) Ostwald viscometer:

A Capillary-tube viscometer has a fluid reservoir to hold a specified volume of sample liquid and of a capillary tube. The hydraulic head of the fluid causes the liquid to flow through the capillary. A clock for measuring the efflux time of the fixed liquid volume and a thermostatic device is also present there.

According to the Hagen—Poiseuille, the pressure drop in a Newtonian liquid passing through a capillary tube is directly proportional to its absolute viscosity if the flow rate is maintained constant.

$$\mu = \frac{\pi R^4}{8VL} (P_1 - P_2) = K \frac{d^4 P}{VL} \quad \dots (1)$$

where, P = Pressure drop across the capillary tube

K = a constant

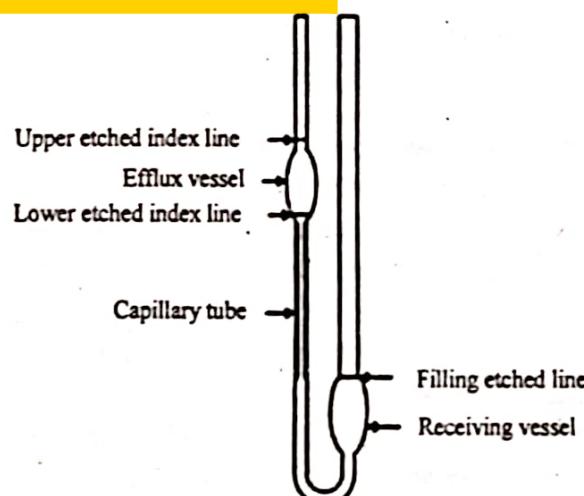


Fig: Ostwald viscometer

The capillary-tube viscometer also gives kinematics viscosity in stokes from measurements of the pressure gradient and of the volumetric flow rate in a cylindrical tube of precisely known dimensions as stated by the above law for the flow of fluids through a capillary:

$$\nu = \frac{\mu}{\rho} = \frac{\pi g h R^4 t}{8 V L} \quad \dots (2)$$

where ν = Kinematics viscosity, stokes (cm^2/sec)

μ = Absolute viscosity, poises ($\text{dyne-sec}/\text{cm}^2$)

ρ = Density of liquid gm/cm^3

g = Acceleration due to gravity, cm/sec^2

h = Vertical distance between ends of capillary, cm

R = Radius of capillary, cm

L = Length of capillary, cm

V = Volume of liquid flowing, cm³, in time ' t ' secs.

t = Time in secs for the volume ' V ' to flow.

The simplest kind of capillary viscometer, the Ostwald viscometer. Various modifications of the classical Ostwald device are available to suit-various application needs. In practice, a sample liquid of fixed volume is charged to the lower receiving vessel and the viscometer is placed in a thermostatic bath. After allowing time for reaching thermal equilibrium, the sample is drawn up into the efflux vessel by suction until the level is above the upper etched index line. The fluid is then permitted to flow down through the capillary by releasing the suction. When the fluid surface passes the upper etched index line, a stop watch is started. The watch is stopped when the surface passes the lower etched index line of the efflux vessel. From this efflux times (t), the kinetic viscosity of the fluid is calculated by multiplying it by the viscometer calibration constant. Equation 2 becomes, $v = At$

$$\text{where } A = \frac{\pi g h R^4}{8 V L} = \text{Viscometer}$$

g) Oxygen analyser using Zirconium oxygen probe:

The flue gas analysis for percentage oxygen using paramagnetic analyser discussed above is carried out by extracting a sample, filtering, cooling and drying it before passing it to the analyser proper. Frequency maintenance is necessary to prevent sampling system blockage. Specialized skills are required to keep it in operation and for calibration. Solid state zirconia sensor is designed around rugged non-sampling techniques to withstand the flue gas environment which is known as in-situ sampling.

The ceramic material zirconium oxide (ZrO_2) when heated, acts as a permeable membrane to oxygen. In other words, the zirconium oxide ceramic sensing element is a closed-end tube which when hot (= 700°C) conducts oxygen ions. Porous platinum coating on the inside and outside of the tube serve as electrodes. When the two electrodes are in contact with gases having different levels of partial pressure; a voltage is produced depending on the ratio of partial pressure satisfying the following equation.

$$E = \frac{RT}{4F} \log \frac{P_1}{P_2} + C$$

where E = Output voltage from the cell

R = Gas constant

P_1 = Partial pressure (concentration) of oxygen in one side

[Reference gas side. Normally air is used]

P_2 = Partial pressure (concentration) of oxygen in flue gas.

C = Cell constant

T = Absolute temperature of the cell

F = Faraday's constant.

Fig. 1 shows a zirconia element equipped with platinum electrodes, thermostatic heating and a source of air as reference gas. The other side of the probe is exposed to the flue gas. The oxygen partial pressure on the reference side of the cell is known and hence the emf produced by the cell indicates the oxygen content of the flue gas.

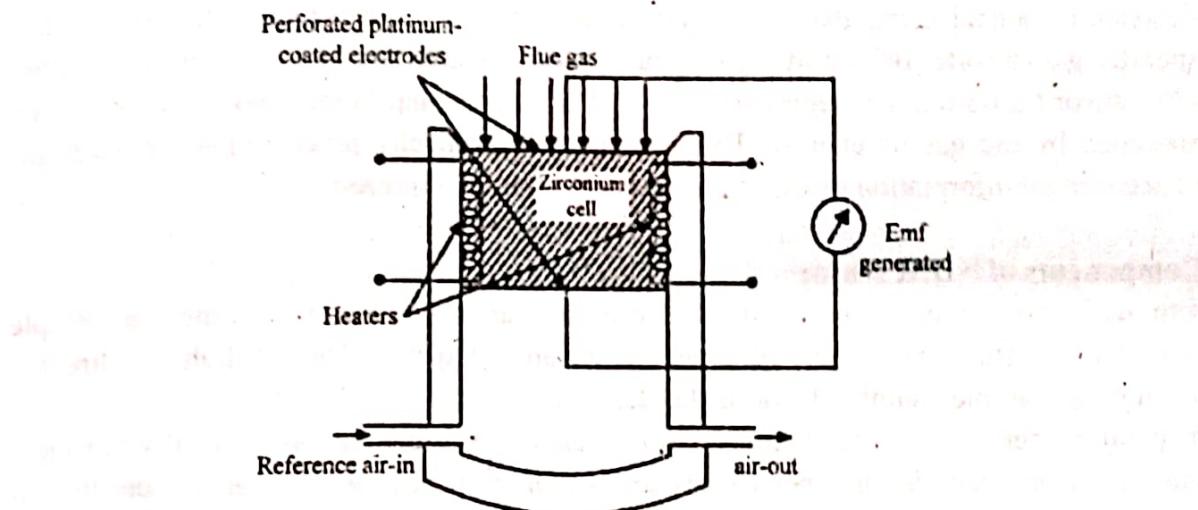


Fig: 1 Zirconium oxide probe

The most common form of the zirconia analyser comprises a probe-mounted zirconia element inserted into the flue duct. In some cases a porous filter is used to protect the element. The probe tip is heated to high temperatures ($\geq 700^{\circ}\text{C}$) by heaters. The electrical signal from the probe is taken to a meter or to an electronic unit where O_2 concentration is calculated and read. Calibration facility and self-diagnostics are incorporated in microprocessor-based electronic units.

The analyser does not have any gas sampling portion and hence needs little or no maintenance. It responds quickly and provides stable measuring for longer periods of time.

h) Flame ionisation detector:

Refer to Question No. 12 of Long Answer Type Questions.

i) NDIR:

A non-dispersive infrared sensor (or NDIR sensor) is a spectroscopy based gas detector. It measures the concentration of constituent gasses in a sample. Each constituent gas in a sample will absorb some infra red at a particular frequency.

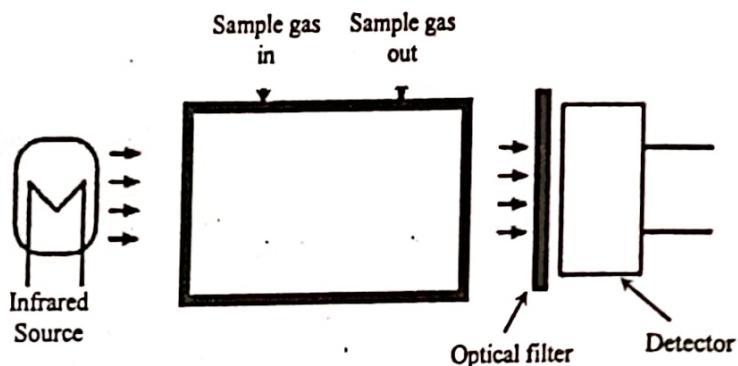
An infra-red beam is allowed to pass through a sample cell. The detector measures the amount of infra-red absorbed by each individual constituent in the sample at the specific wavelengths. A NDIR detector measures the volumetric concentration of the gasses in the sample.

It is non-dispersive in the sense of optical dispersion. The infrared energy is allowed to pass through constituent specific optical filters. No prism or grating is used for IR dispersion. Non-Dispersive Infrared (NDIR) technology utilizes a broadband infrared (IR) emitter, which covers all of the wavelengths of interest for a given set of gases to be measured. Optical Band Pass filters allow that portion of IR wavelengths at which a specific gas absorbs IR energy. An infrared sensor is attached to each band pass filter. The sensor transduces an electrical signal that is proportional to the amount of IR energy absorbed by the gas of interest. This signal is electronically processed to develop gas concentration information reported in engineering units of interest.

Components of NDIR sensor

The main components of an NDIR sensor are an infrared source (lamp), a sample chamber or light tube, a light filter and an infrared detector. The IR light is directed through the sample chamber towards the detector.

In parallel there is another chamber with an enclosed reference gas, typically nitrogen. The gas in the sample chamber causes absorption of specific wavelengths according to the Beer-Lambert law, and the attenuation of these wavelengths is measured by the detector to determine the gas concentration.



The detector has an optical filter in front of it that eliminates all light except the wavelength that the selected gas molecules can absorb.

Ideally other gas molecules do not absorb light at this wavelength, and do not affect the amount of light reaching the detector however some cross-sensitivity is inevitable. For instance, many measurements in the IR area are cross sensitive to H₂O so gases like CO₂, SO₂ and NO₂ often initiate cross sensitivity in low concentrations.

Gases and their sensing wavelengths

- O₂ - 0.763 μm
- CO₂ - 4.26 μm, 2.7 μm, about 13 μm
- carbon monoxide - 4.67 μm, 1.55 μm, 2.33 μm, 4.6 μm, 4.8 μm, 5.9 μm
- NO - 5.3 μm, NO₂ has to be reduced to NO and then they are measured together as NOx; NO also absorbs in ultraviolet at 195-230 nm, NO₂ is measured at 350-

- 450 nm; in situations where NO_2 content is known to be low, it is often ignored and only NO is measured; also, 1.8 μm
- NO_2 - 6.17-6.43 μm , 15.4-16.3 μm , 496 nm
- N_2O - 7.73 μm (NO_2 and SO_2 interfere), 1.52 μm , 4.3 μm , 4.4 μm , about 8 μm
- HNO_3 - 5.81 μm
- NH_3 - 2.25 μm , 3.03 μm , 5.7 μm
- H_2S - 1.57 μm , 3.72 μm , 3.83 μm
- SO_2 - 7.35 μm , 19.25 μm
- HF - 1.27 μm , 1.33 μm , etc.

Applications of Non-dispersive Infrared Spectroscopy

Environmental Applications

Environmental applications include monitoring of emissions from vehicles, industrial sites and the measurement of other trace atmospheric gases.

Vehicle Gas Emission Monitoring

Vehicles emit a range of pollutants into the atmosphere including carbon dioxide, carbon monoxide, nitrogen oxides, volatile organic compounds (VOC) and poly-aromatic hydrocarbons (PAH's). These pollutants are harmful to human health and local flora and fauna. Nitrogen oxides emitted from vehicles are responsible for the production of photochemical smog in many major cities around the world.

Industrial Applications – Nuclear Fuels

One of the most important quantities that specify the physico-chemico state of a uranium-plutonium mixed oxide (MOX) fuel pellet which is the oxygen-to-metal atomic ratio (O/M ratio).