LIQUID ANALYSIS

Multiple Choice Type Questions

1. The constant polarizing voltage applied in polarographic cell is [WBUT 2009, 2012]

a) 0.5V b) 0.8V c) 1.5V d) 2.0V

Answer: (b)

2. Consider a solution containing NaCl in contact with pure water, following happens [WBUT 2010]

a) Cl– ion has greater mobility b) Na+ ion has greater mobility

c) no ions will be formed

Answer: (b)

3. Viscosity of a non-Newtonian liquid is known as [WBUT 2010]

a) kinematic viscosity b) specific viscosity

c) consistency d) relative viscosity

Answer: (c)

4. Which viscometer is used for both Newtonian and non-Newtonian fluids? [WBUT 2010, 2013, 2017]

a) Saybolt viscometer b) Ostwald viscometer

c) Cone and Plate viscometer d) none of these

Answer: (c)

5. Chemical formula of TMS is [WBUT 2011]

a) $(CH_3)_4$ Si b) $(CH_4)_3$ Si c) CH_3Si_4 d) $(CH_3)_4$ Si_3

Answer: (a)

6. Dropping mercury electrode is used in [WBUT 2011, 2013, 2014]

a) conductivity test b) polarography

c) pH measurement d) dissolved oxygen analysis

Answer: (b)

7. A buffer may be defined as a solution which maintains a constant value of [WBUT 2011, 2014, 2016]

a) density b) pH c) viscosity d) none of these

Answer: (b)

8. A buffer solution is a solution that [WBUT 2013]

a) retains its pH for a long time

b) cannot retain its pH for long

c) has no electrolytic

d) acts as an intermediate solution between two solutions of different pH

Answer: (a)

9. Saturated KCl is used in salt bridges for measuring pH of a liquid because [WBUT 2015]
   a) K^+ and Cl^- have very large difference in mobilities
   b) K^+ and Cl^- have similar mobilities
   c) resistivity change is very little with applied voltage
   d) it has high viscosity

Answer: (b)

10. Viscosity of the non-Newtonian liquid is known as [WBUT 2016]
   a) kinematic viscosity b) specific viscosity
   c) consistency d) relative viscosity

Answer: (c)

11. Example of efflux type viscometer is [WBUT 2016]
   a) capillary viscometer b) saybolt viscometer
   c) searle's rotating viscometer d) none of these

Answer: (b)

12. Unit of kinematic viscosity is [WBUT 2016]
   a) stokes b) poise c) Rhe d) none of these

Answer: (a)

13. The resistance of a glass electrode in a pH meter may be as high as [WBUT 2017, 2018]
   a) 10^5 ohm b) 10^8 ohm c) 10^{15} ohm d) 10^{20} ohm

Answer: (b)

Short Answer Type Questions

1. What are the advantages and disadvantages of Quinhydrone electrode? Write down the emf equation of this electrode. [WBUT 2009]

Answer:

The advantages and disadvantages of Quinhydrone electrodes are-

Advantages:

- The electrode can be quickly set up by adding a pinch of quinhydrone to the solution and dipping a clean platinum wire into it.
- a small quantity of the sample liquid is required for the measurement
- It indicates accurate pH values even in the presence of interfering osidising ions.

Disadvantages:

- It cannot be used Fe^{2+}, MnO_2, aniline, etc. which react with quinine or hydroquinone.

- Hydroquinone begins to oxide at pH = 8.5, and therefore, it cannot be used for alkalis having pH ≥ 8.5.

An inert electrode(such as platinum) when immersed in the solution, the emf E developed by it can be given by using the Nernst equation which is-

$$E = E^0 + \frac{2.303RT}{nF}\log\frac{[QH_2]}{[Q][H^+]^2}$$

$$= E^0 + \frac{2.303RT}{2F}\{\log\frac{[QH_2]}{[Q]} - 2\log[H^+]\}$$

The rario $[QH_2]/[Q]$ can be maintained at 1 by saturating the solution by quinhydrone, that is 1:1 molar compound of quinine and hydroquinone

Now, the above equation becomes, $E = E^0 + 0.591pH$

The standard oxidation potential of quinhydrone electrode is -0.6994V at 25°C

2. For measuring Cu^{2+} concentration in water sample, the concerned ORP cell considered of a silver electrode dipped into 1 M $AgNO^3$ solution connected by a salt bridge to another half-cell consisting of copper electrode dipped into the sample water. The measured cell potential with the copper serving as anode was 0.62 V at 25°C. Find the concentration of Cu^{2+} in the sample. Given, standard reduction potentials of Ag^+ and Cu^{2+} are 0.80 V and 0.34 respectively. [WBUT 2010]

Answer:

Copper was oxidized because it served as anode. Therefore Ag^+ was reduced. So, the redox reaction was

$$Cu(s) + 2Ag^+(aq) + 2Ag(s)$$

Given, $E_{cell} = 0.62V$ $\quad E^\circ_{cathode} = 0.80V$

$E^\circ_{anode} = 0.34V$ and $n = 2$, therefore,

$E^\circ_{cell} = (0.80 - 0.34)V = 0.46V$

So, we get

$$E_{cell} = E^\circ_{cell} - \frac{0.0592}{2}\log\frac{[Cu^{2+}]}{[Ag^+]^2}$$

$$= 0.46 - 0.0296\log\frac{[Cu^{2+}]}{(1)^2}$$

or, $\log\frac{[Cu^{2+}]}{1} = -5.4$

Hence,

$$[Cu^{2+}] = \text{anti}\log(-5.4)$$

$$= 3.92 \times 10^{-6} M$$

3. Mention some applications of Polarograph. **[WBUT 2011]**

Answer:

Applications of Polarography

- Polarographic methods can be used for analyzing a wide range of materials. In metallurgy, Cu, Sn, Pb, Fe, Ni, Zn, Co, Sb, and Bi can be determined in light and zinc-based alloys, copper alloys, and aluminum bronze; the control of effluents is often carried out using polarographic methods.
- Cyanide concentrations down to ~0.1 ppm can be determined, and sludges and sewage samples as well as fresh and sea waters can be analyzed.
- Trace and toxic elements can be determined polarographically in foodstuffs and animal feed, in soils, and in pharmaceutical products.
- Some compounds are themselves polarographically reducible or oxidizable – for example, ascorbic acid, riboflavin, drugs such as phenobarbitone and ephedrine, and substances such as saccharine. Body fluids, plastics, and explosives can also be analyzed by polarographic techniques.

4. What is pH? Explain the principle of pH measurement. **[WBUT 2012]**

Answer:

1st part:

pH can be viewed as an abbreviation for power of Hydrogen ion concentration. pH is a measure of the acidity or alkalinity of a water solution.

The mathematical definition of pH is a bit less intuitive but in general more useful. It says that the pH is equal to to the negative logarithmic value of the Hydrogen ion (H^+) concentration, or

$pH = -log\ [H^+]$

pH can alternatively be defined mathematically as the negative logarithmic value of the Hydroxonium ion (H_3O^+) concentration. Using the Bronsted-Lowry approach

$pH = -log\ [H_3O^+]$

2nd part:

pH measurement is based on the use of a pH sensitive electrode (usually glass), a reference electrode, and a temperature element to provide compensation due to temperature variation to the pH analyzer. The pH electrode uses a hydrogen ion sensitive glass in contact with the solution, which develops a potential (voltage) relative to the reference electrode proportional to the pH of the solution. The reference electrode is designed to maintain a constant potential at any given temperature, and serves to complete the pH measuring circuit within the solution. It provides a known reference potential for the pH electrode. The difference in the potentials of the pH and reference electrodes provides a millivolt signal proportional to pH. Most pH sensors are designed to produce a 0 mV signal at 7.0 pH, with a (theoretically ideal) slope (sensitivity) of -59.16 mV / pH at 25°C.

5. What is polarization? How can it be minimized or eliminated?
[WBUT 2013, 2014, 2018]

Answer:
Polarization is a mechanism that typically results in a change in the potential of an electrode during electrolysis, when the anode's potential becomes nobler than that of the cathode. It has the effect (based on conditions) of decreasing the output voltage of batteries, increasing the voltage required for electrolysis cells or lowering currents.
Polarization can also be described as a kinetic deviation from equilibrium due to an electric current passing through a galvanic cell. When electrode reactions take place, the potential will no longer be at equilibrium due to current flow through an electrochemical cell, which causes a change in the electrode potential. This electrochemical phenomenon is termed Polarization. Polarization of anode is called anodic polarization and Polarization of cathode is called as cathodic polarization. A cell or electrode is said to be polarized when there is little or no change in current with larger changes in potential.
The phenomenon of electrochemical polarization may be both harmful and beneficial. While electrochemical polarization causes a loss of electric power in electrolysis and reduces the efficiency of galvanic cells, it also has the capacity to inhibit certain undesirable corrosive processes.

In order to minimize the effect of polarization:
1. Platinum electrode is to be coated with platinum black.This serves to reduce the polarization or IR-drop at the electrode electrolyte interface.
2. To eliminate electrode by employing the technique of electrode less cell
3. In case of amperometric measurements, the electrodes are to be powered by ac source.

6. What do you mean by buffer solution? Name various types of electrodes used for pH measurement. Write down Nernst equation for Redox reaction. [WBUT 2017]
Answer:
1st Part:
A buffer solution is one whose pH remains unaltered when small quantities of an acid or an alkali are added to it. It is a *solution* in water of a mixture of a weak acid or base and its salt.

2nd Part: ***Refer to Question No. 3(a) (1st part) of Long Answer Type Questions.***

3rd part:
The Nernst equation is an equation that relates the reduction potential of an electrochemical reaction to the standard electrode potential (E^0), temperature (T in Kelvin), and concentrations [Red] and [Ox] of the chemical species undergoing reduction (Red) and oxidation (Ox).

$$E = E^0 - \frac{kT}{ne} \ln \frac{[\text{Red}]}{[\text{Ox}]}$$

$$= E^0 - \frac{RT}{ne} \ln \frac{[\text{Red}]}{[\text{Ox}]}$$

$$= E^0 - \frac{RT}{ne} \ln Q_r$$

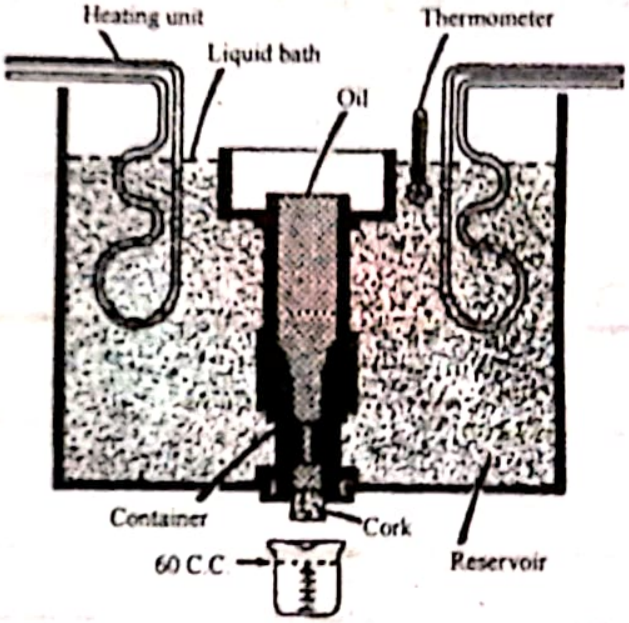
7. What are the advantages and disadvantages of Quinhydrone electrode? **[WBUT 2018]**

Answer: *Refer to Question No. 1 of Short Answer Type Questions.*

8. Describe a Saybolt viscometer with proper diagram. **[WBUT 2018]**

Answer:

Saybolt Viscometer is a device used to measure the viscosity of a fluid. It measures the time taken by the fluid to fill a 60cc of the container by controlling the heat of the fluid.



Efflux cup viscometers are most commonly used to measure the viscosity of oils, syrups, varnish, paints and Bitumen emulsions.

It contains a thermostatically controlled bath to hold liquid and orifice which are under tight temperature control. Temperature is read by a thermometer. The sample liquid is placed in a separate chamber and connected to the orifice assemble as shown in the diagram. There are three types of orifices available-Universal, Furol, Asphalt. The furol and asphalt orifices, respectively, have an efflux time of approximately, one-tenth and one-hundredth that of the universal orifice. A cup of standard volume is placed to collect the sample fluid that comes out from the orifice. The cup orifice combination should be selected to provide an efflux- time within the range of 20 to 100 seconds. Of these types, the universal orifice (Saybolt universal viscometer) is most commonly used and its efflux time is designated as Saybolt universal seconds. It measures the time required for 60 cc of sample fluid to flow out through an orifice having dimensions of 0.176 cm in diameter

and 1.225 cm in length. Saybolt universal seconds (t) can be converted to kinematic viscosity (v) by the following equations:

When $t < 100$ secs, $v = 0.226t - \frac{195}{t}$ Centistokes

When $t > 100$ secs, $v = 0.220t - \frac{135}{t}$ Centistokes

Long Answer Type Questions

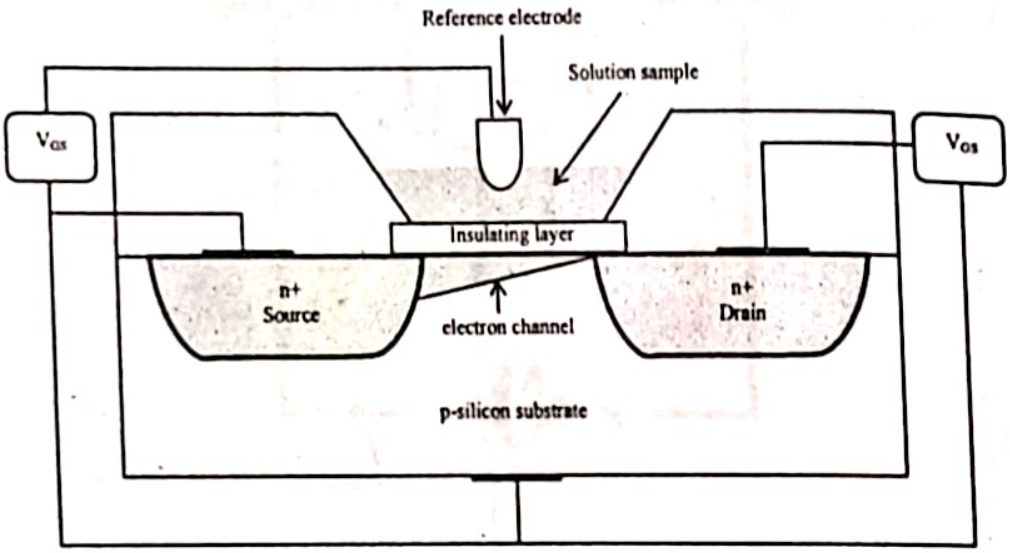
1. a) Draw the schematic of the Ion Selective FET (ISFET) electrode and describe the working of the same. How can selectivity and reliability be improved of the ISFET? [WBUT 2009, 2015, 2016]

b) What do you mean by concentration polarization? [WBUT 2009, 2012, 2015]

Describe the linear-scan polarography process with necessary circuit diagram. Also explain the nature of the polarograph found. [WBUT 2009, 2016]

Answer:

a)



The above diagram shows the ISFET electrode structure. A normal FET is a three terminal device in which current can flow from source to gate is modified by a gate terminal. But in case of a ISFET sample solution with a reference electrode is placed in place of the metallic gate and gate oxide of the normal FET. An insulating layer is also present in contact with the solution for detecting a specific analyte. This insulating layer defines the functionality and sensitivity of the ISFET sensor. The potential between the solution and the ion selective material is proportional to the ion activity in the test solution. Most applications use the ISFET in conjunction with a separate reference electrode; some applications have a reference electrode into the body of the ISFET.

The selectivity of the ISFET can be varied via the addition of ion-sensing membranes, hus allowing also the detection of other species.

The ion selectivity is achieved by adding ionophores to the membrane material.

b) When electrolysis occurs in a solution, one electrode may show a constant potential while at the other electrode concentration polarization occurs. The voltage current characteristic graph of such a system known as polarograph.

Concentration polarization means

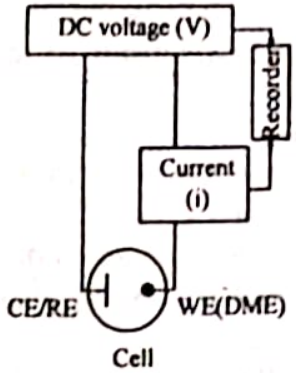
- Accumulation of excess particles in a thin layer adjacent to the substrate.
- Adsorption or filtration on/by substrate.
- Increases resistance to solvent flow.

When a potential applied that is sufficient to oxidize or reduce an analyte of the electrode surface such that the redox reaction is fast in comparison with mass transfer, the analyte is quickly consumed at the electrode surface so that its concentration become very small with respect to its value in bulk solution. This effect is known as ***concentration polarization*** and results in a concentration gradient where the amount of analyte at the electrode surface is much lower than in bulk solution.

Linear-scan polarography was first type of voltametry to be discovered and used. There is essentially no convection or migration, a dropping mercury electrode (DME) is used as the working electrode. Because there is no convection, diffusion alone controls polarographic limiting currents.Compared with hydrodynamic voltammetry, polarographic limiting currents are an order of magnitude or more smaller since convection is absect in polarography.

Linear scan polarography is a form of DC polarography. In simple DC polarography the potential applied is linearly varying voltage called ramp and the resulting current response is sigmoid in shape.

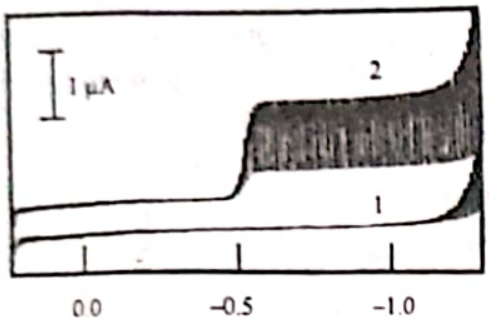
A typical example of the above is given in the figure.



Schematic circuit of a polarographic circuits for two electrode

WE - Working electrode (DME-Dropping mercury electrode)
RE - Reference electrode
CE - Counter electrode
DC (V) - DC voltage source
Current (i) - Current measuring device

The figure below shows the current-potential curves obtained in 0.1M HCl and 0.1M HCl + 1.0mM Cd^{+2}. Here 0.1M HCl is the supporting electrolyte and 1.0 mM Cd^{+2} is the supporting electrolyte and 1.0mM Cd^{+2} is the electroactive species that is reduced or oxidized at the electrode. Here current is measured without damping the current oscillation.



An example of a DC polarogram recorded without damping. Polarogram 1 in 0.1M KCl supporting electrolyte and 2 in 1.0mM Cd^{2+} + 0.1MHCl

2. What is pH of a solution? How pH is measured with a glass electrode? Explain the working of calomel and Ag/AgCl reference electrodes. Mention a few applications where pH measurement and control are required. **[WBUT 2011, 2015, 2018]**

OR,

What do you mean by pH? Explain the working of Calomel reference electrode in the process of pH measurement. **[WBUT 2013]**

OR,

List the types of electrode used for pH measurement. Explain construction details of any one of them. **[WBUT 2013]**

OR

Write short note on Measurement of pH using glass electrode **[WBUT 2016]**

OR,

What do you mean by pH of a solution? Explain the operation of a Calomel reference electrode used in pH measurement with suitable diagram. **[WBUT 2016]**

Answer:

1st part:

pH of a solution is a measure of the acidity or basicity of an aqueous solution. Solutions with a pH less than 7 are said to be acidic and solutions with a pH greater than 7 are basic or alkaline. Pure water has a pH very close to 7.

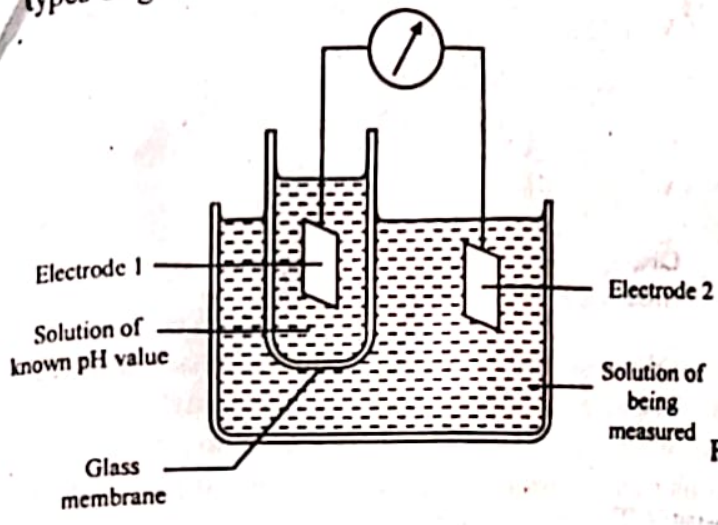
pH is defined as the decimal logarithm of the reciprocal of the hydrogen ion activity, a_H+, in a solution $pH = -\log_{10}(a_{H^+}) = \log_{10}\left(\frac{1}{a_{H^+}}\right)$

2nd part:

Glass electrode

The glass electrode consists of a glass probe containing two electrodes, a measuring one and a reference one, separated by a solid glass partition. Neither of the electrodes is in fact glass. The reference electrode is a screened electrode, immersed in a buffer solution, which provides a stable reference e.m.f. that is usually 0V. The tip of the measuring electrode is surrounded by a pH-sensitive glass membrane at the end of the probe, which permits the diffusion of ions according to the hydrogen ion concentration in the fluid outside the probe. The measuring electrode therefore generates an e.m.f. proportional to

pH that is amplified and fed to a display meter. The characteristics of the glass electrode are very dependent on ambient temperature, with both zero drift and sensitivity drift occurring. Thus temperature compensation is essential. This is normally achieved through calibrating the system output before use by immersing the probe in solutions at reference pH values. Whilst being theoretically capable of measuring the full range of pH values between 0 and 14, the upper limit in practice is generally a pH value of about 12 because electrode contamination at very high alkaline concentrations becomes a serious problem and also glass starts to dissolve at such high pH values. Glass also dissolves in acid solutions containing fluoride, and this represents a further limitation in use. If required, the latter problems can be overcome to some extent by using special types of glass.



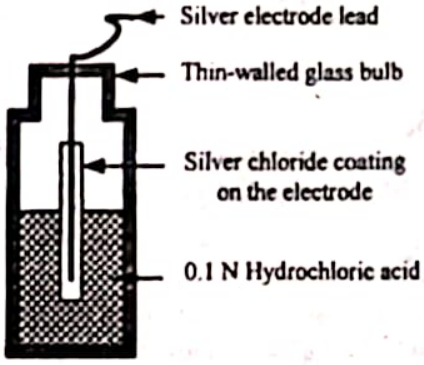


Fig: Details of typical glass electrode

3rd part:

Calomel Electrode

Calomel electrode (Fig. (i)) contains a pool of mercury at the bottom of a glass tube. On top of mercury, there remains a paste of mercury and mercurous chloride (calomel) in potassium chloride solution. The electrolyte is also a solution of potassium chloride. A platinum wire, which may be amalgamated, maintains contact with the mercury pool.

The calomel electrode serves as a secondary standard reference electrode. The salt bridge serves as a connector between the reference electrode and the measuring (or indicator) electrode. It consists of KCl solution concentration 3.8 M with excess gelatin and plugged at both ends with a porous fritted glass stopper. The oxidation potential of the Hg/Hg_2Cl_2 electrode is – 0.2415 V for saturated KCl solution at 25°C.

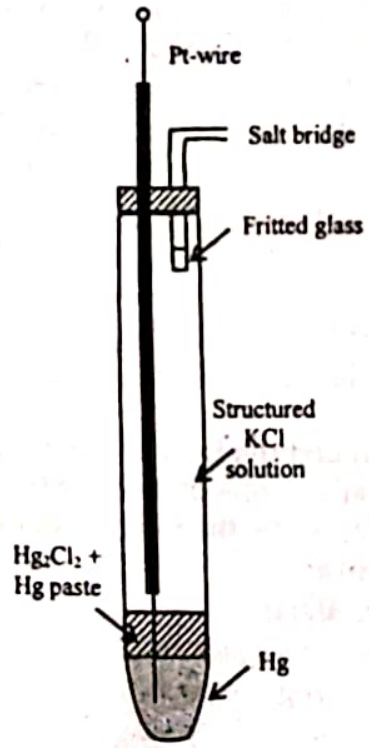


Fig: (i) Calomel electrode

Silver / silver chloride electrode

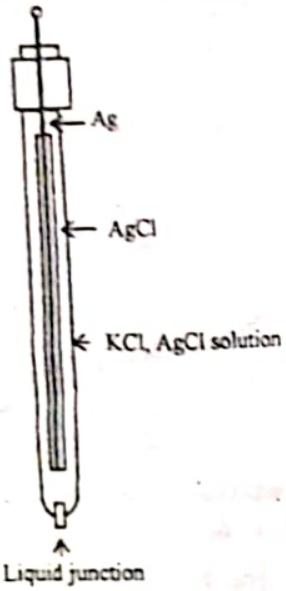


Fig: (ii) Ag/AgCl electrode

Silver / silver chloride electrode has the advantage that it is (i) reversible, (ii) stable, and it can be combined with cells containing chlorides without inserting salt bridges.

It consists of a silver electrode coated with silver chloride and dipped in a saturated solution of potassium chloride and silver chloride [Fig. (ii)]. A liquid junction separates it from the process fluid.

The Ag/AgCl electrode acts as a secondary standard reference electrode having a standard potential of – 2.2224 V at 25°C.

4th part:

pH measurement mainly required for the purpose of finding the chemical characteristics of a solution, apart from that it is an essential first step towards managing chemical reactions. Nearly all industries that deal with water – not merely chemical industries, but also agriculture and fishery-related industries, biological industries, public organizations – need pH measurement at some stage or other.

- In textiles, dyeing, paper and pulp industries, pH measurement is necessary in the desulphurization process.
- In metallurgy, when extracting a particular material from crude ore or mixed metal, pH is controlled so as to extract only the desired metal without dissolving the slag.
- In electrochemical industries, pH control is necessary in plating, etching of metal surfaces and the manufacture of batteries. For example, without proper control of the pH of the plating solution, the finished plating will lack the optimum colour and lustre or is likely to peel off.

3. a) List the type of electrodes used for pH measurement. Explain the construction details of one of them. Why is reference electrode required for pH measurement?
b) Describe the method of dissolved oxygen analysis. **[WBUT 2014]**

Answer:

a) 1st Part:

The various electrodes used for pH measurements are as follows:

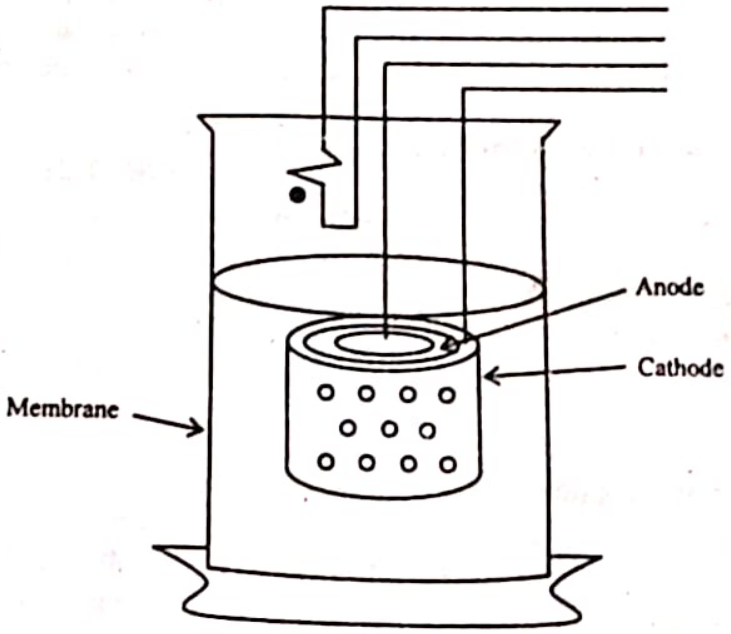
1. Hydrogen electrode
2. Calomel electrode
3. Quinhydrone and antimony electrodes
4. Glass electrodes.

2nd Part: ***Refer to Question No. 2 of Long Answer Type Questions.***

3rd Part:

Glass electrode provides the half –cell potential only and it is not possible to measure the absolute value of this potential. So reference electrode is used to have a reference half cell potential. The half cell potential from the glass cell is measured relative to the reference half cell potential to show the pH value.

b) A typical electrochemical sensor probe is a membrane electrode which is used for measuring oxygen dissolved in water. Both galvanic and polarographic probes are available. In conformity with the sequel of the text, a typical galvanic type is discussed here in brief. It has an inner cylindrical anode often made of lead and outer perforated cathode usually of silver – the combination being immersed in an aqueous solution of $KHCO_3$ which is held in position by a silicone rubber membrane properly arranged as shown if figure. This membrane is permeable to oxygen but not to water or other ions likely to be present. Diffused oxygen is reduced at the cathode as per the equation: $O_2+H_2O+4e^- \rightarrow 4OH^-$ and a current proportional to the partial pressure of oxygen is obtained. With change in temperature, however, this current changes at a rate 6%/°C and a compensation circuit is, therefore, necessary which requires that the temperature is continuously measured/monitored by a thermistor/RTD as shown. The probe has a wide range of measurement starting from about 5 to 10 mg/litre to the saturation value. The anode gets eroded on continuous use, the rate of erosion being dependent on the amount of oxygen measured and the initial size and shape of the electrode. It, therefore, is required to be replaced after specified periods of time.



A dissolved oxygen analysis cell

4. Describe the operation of the crystal oscillator method of moisture measurement. **[WBUT 2014]**

Answer:

A typical scheme is shown in figure below. A 9 MHz dry crystal is chosen whose frequency drops when exposed to the sample. The difference frequency Δf is converted into a voltage, amplified, detected and then displayed. The two crystals shown are usually exposed to two gases alternately for equal intervals of time of about 30 sec, so that their contamination becomes identical and stability is frequency output is thus ensured. It can measure a humidity of about 1 to 300 V ppm. A programmable microprocessor is used for providing the up-to-date display. It is used to measure low amount of moisture content is gases like hydrogen, ethylene, refrigerants and natural gas. It is, however, a costly instrument.

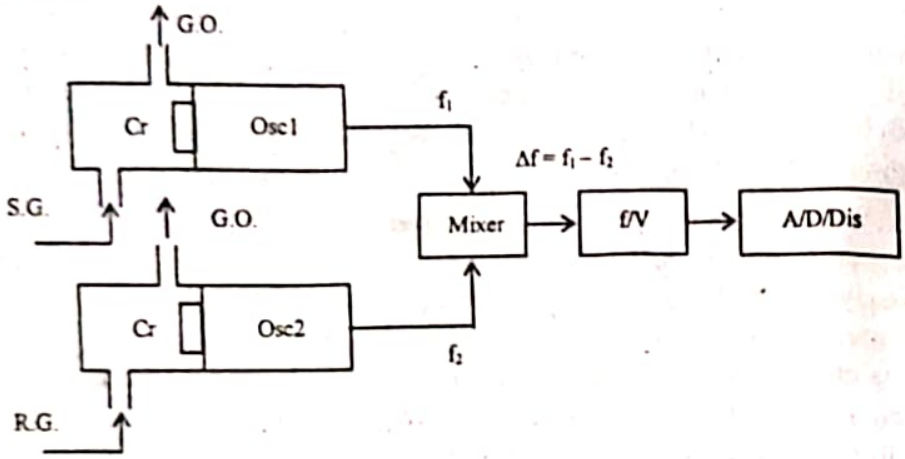


Fig: Hygrometer based on quartz crystal oscillator (f/V frequency to voltage converter, A/D/Dis amplifier, detector and displays, Cr crystal, G.O. gas out, S.G. sample gas, R.G. reference gas

5. Write short notes on the following:

a) Colorimetry **[WBUT 2010, 2013, 2014, 2018]**

OR,

Colorimeter **[WBUT 2012]**

b) Dropping mercury electrode **[WBUT 2012]**

c) Karl-Fischer Regent **[WBUT 2015]**

d) Nebulizer **[WBUT 2018]**

Answer:

a) Colorimetry:

Color is a very subjective phenomena, causing the description of color difference or the comparison of two colors to be quite difficult. However, there are methods that have been developed that allow for the description of color to be an objective matter rather than a subjective matter. Giving color description a quantitative nature is the focus of a field called colorimetry. The Reynolds group utilizes in-situ colorimetric analysis in the study of electrochromic polymers and devices. This method allows us to graphically represent the path of color change quantitatively during electrochromic switching.

The word colorimetry is derived from Latin: color = colour and Greek: metron = measure. It is the science of measuring colour. There are broadly two types of colorimetry, the measurement of surface colours, such as painted metal, that are illuminated by incident light; and the measurement of light emitting objects, such as lamps.

Colorimeters typically consist of three light detectors, each with a spectral response corresponding to the tristimulus receptors (cones) of the human eye. The combined output of these detectors gives chromaticity values.

A typical and simplified arrangement of colorimetry is shown below.

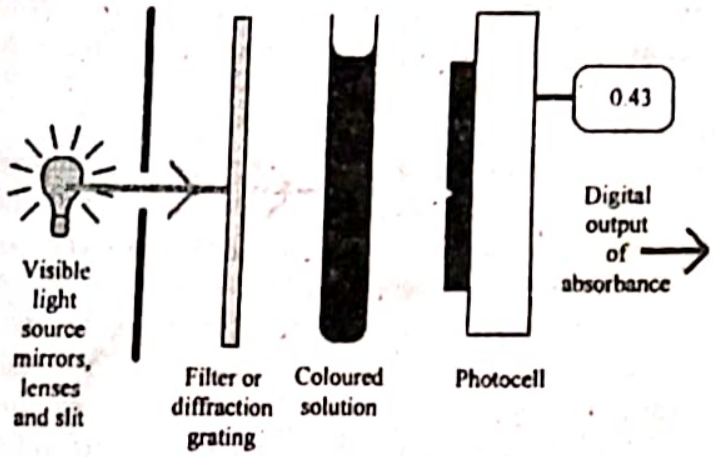


Fig: A simplified diagram of a colorimet

In the above arrangement, light from a suitable source is passed through a light filter to select the most appropriate wavelength of light, some of which is then absorbed by the solution held in a special glass cuvet (a sort of 'test tube').The amount of light absorbed is called, and measured as, the absorbance which is a function of the coloured solute concentration. The filter is chosen to select the band of wavelengths which are most strongly absorbed by the coloured solution

b) Dropping mercury electrode:

Working principle: The dropping mercury electrode consists of a length of marine barometer tubing with a fine capillary and a head of mercury above it. Mercury, usually under force of gravity, is forced through a section of very fine glass capillary. A mercury drop starts, grows and finally falls off as another drop starts. The measured current will naturally tend to follow this process of increasing steadily, dropping sharply and finally increasing again. The mercury head is so adjusted that it gives a drop time of 2-5 s. The head is generally kept between 40 and 80 cm. The internal diameter of the capillary tube is of the order of 0.03-0.05 mm and the length of the capillary is about 8 cm. A platinum wire is immersed in the mercury reservoir and the dropping mercury electrode is coupled with an unpolarized electrode. This electrode is useful over the range form 0.4 to -2.8 V; referred to the normal hydrogen electrode. Above 0.4 V, mercury dissolves and gives an anodic wave. At potentials more negative than -1.5 V, the electrolytes begin to discharge.

The main advantages of dropping mercury electrode are:

- Hydrogen has a much larger over-potential mercury. It renders possible the deposition of substances difficult to reduce, as for example, the alkali ions.
- It provides nearly ideal conditions for obtaining a diffusion-controlled limiting current, which is reproducible.
- It provides a continuously refreshed surface, which is conducive to a high degree of reproducibility for the current measurements. The constant renewal of the electrode surface eliminates passivity or poisoning effect.

But, it cannot be used for dilutions of less than 10^{-5} M, due to the presence of a relatively large charging current. Nevertheless, high sensitivity derivative instruments may be 200 times more sensitive, as they compensate for the effect of charging current.

c) Karl-Fischer Regent:

The Karl Fischer is a titration method uses Karl Fischer reagent, which reacts quantitatively and selectively with water, to measure moisture content. Karl Fischer reagent consists of iodine, sulfur dioxide, a base and a solvent, such as alcohol. he working medium is the solvent or solvent mixture in which the sample is dissolved and the Karl Fischer reaction takes place. A suitable working medium assures the stoichiometry of the Karl Fischer reaction and must be able to dissolve the sample and the products of the titration reaction as well as allow confident end determination[1]. Only few solvents can fulfill all these requirements. Methanol is the preferred choice for the working medium as it allows for a rapid and stoichiometric course of reaction. Most samples dissolve easily in methanol and it gives a sensitive and reliable indication of the end point. Often, other solvents are added although the methanol content should never fall below 25 %. In the ideal pH range 5-7, the Karl Fischer reaction runs quickly and stoichiometrically. At higher pH values a side reaction occurs which consumes iodine and leads to vanishing end points. In strongly acidic conditions the reaction constant of the Karl Fischer reaction decreases and the course of the titration is slower. n practice, a pH range of 4-7 is acceptable.

The general reactions behind Karl Fischer titration are as follows:

$$CH_3OH + SO_2 + RN \rightarrow [RNH]SO_3CH_3$$

$$H_2O + I_2 + [RNH]SO_3CH_3 + 2\,RN \rightarrow [RNH]SO_4CH_3 + 2[RNH]I$$

(RN = Base)

This reaction consumes water and iodine in a 1:1 ratio. In order to assure a stoichiometric course of the Karl Fischer reaction, certain fundamental requirements must be met and several potential interferences must be avoided. The choice of working medium and pH range are the most critical considerations.

d) Nebulizer:

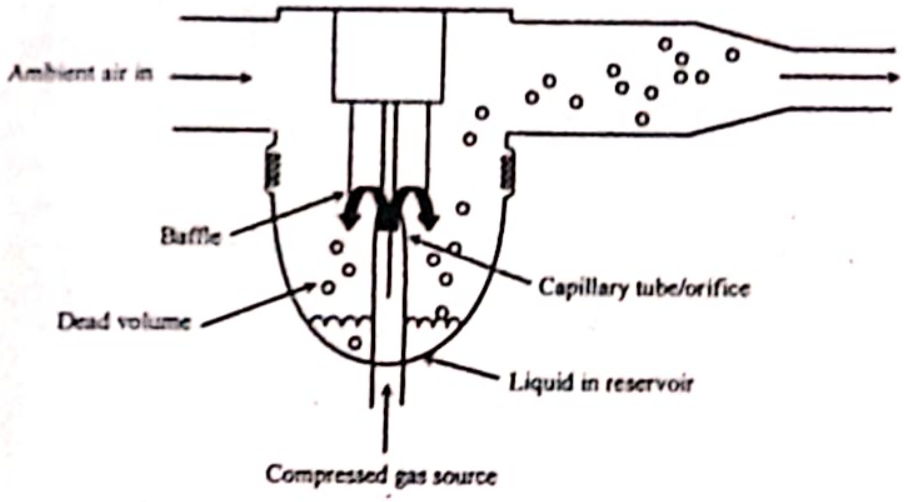
The Nebulizer is a machine that atomizes and vaporizes liquid medication into small aerosol droplets, or a mist, that can be used in therapeutic medicine or analytical instrumentation.

In medicine, a **nebulizer** or **nebulizer** is a device commonly used for the treatment of asthma, cystic fibrosis, COPD and other respiratory diseases. It is used to administer medication in the form of a mist inhaled into the lungs.
Analytical nebulizers are another form of nebulizer that is used primarily in laboratory settings for elemental analysis.

Types of nebulizers
Nebulizers use oxygen, compressed air or ultrasonic power to break up solutions and suspensions into small aerosol droplets. Based on the power used for the process of nebilization, nebulizers can be types as

1. Pneumatic nebulizers
2. Ultrasonic wave nebulizers

A pneumatic nebulizer uses a pressurized gas supply as the driving force for liquid atomization. Compressed gas is allowed to pass through a jet, causing a region of negative pressure. The solution to be aerosolized is entrained into the gas stream and is sheared into a liquid film. A baffle, placed in the aerosol stream, produces smaller particles and allows larger particles to return to the liquid reservoir.
Figure below shows the basic arrangement of a pneumatic nebulizer.



An ultrasonic nebulizer uses a piezoelectric crystal to create a series of ultrasonic sound waves. The crystal is powered by an oscillator with frequency in the range of 30-40 MHz.A driver circuits interfaces the oscillator and the crystal as shown in the figure below.These waves break the liquid particles down into tiny particles to create the aerosol. Like the pneumatic nebulizer a baffle is placed in the aerosol stream. for necessary therapeutic and analytical applications. Ultrasonic nebulizers provide a fog of medicine that has much smaller particles and can travel much deeper into the patient's lungs, resulting in much faster relief. Patients who have asthma, COPD, pneumonia, chronic bronchitis, emphysema, or other chronic conditions, most often use them for medicinal purposes. While other devices can deliver the medicine in a mist form,

ultrasonic nebulizers provide a fog of medicine that has much smaller particles and can travel much deeper into the patient's lungs, resulting in much faster relief.