PHARMACEUTICAL ANALYSIS

UNIT 5 NOTES

- CONDUCTOMETRY
- POTENTIOMETRY
- POLAROGRAPHY

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CONDUCTOMETRIC TITRATION

Conductometric Titration is an electrochemical method of analysis used for the determination of concentration of unknown solution on the basis of its electrical conductivity by means of a **Conductometer.**

PRINCIPLE

- During the titration process, the conductivity of the solution changes due to the replacement or movement of Ions.
- These changes are monitored and plotted against the volume of titrant added.
- The end point is identified as the point where a significant change in conductivity occurs.

SOME IMPORTANT DEFINITIONS

① **Conductance** : Conductance is the ability of a material or solution to conduct electric current. It is reciprocal of resistance and is denoted by symbol G.

$$G = 1/R$$

② **Resistance** : Resistance is the property of a material that opposes the flow of electric current through it. It is denoted by R

$$R \propto l/a \quad \text{or} \quad R = \rho l/a$$

- Here ρ is specific Resistance

③ **Specific Conductance** : It is defined as conductance of a solution measured per unit length & cross sectional area. Also known as 'kappa'.

$$K = \frac{l}{a} G$$

FACTORS AFFECTING CONDUCTIVITY

- Types of Ions
- Concentration of Ions
- Temperature
- Mobility of Ions

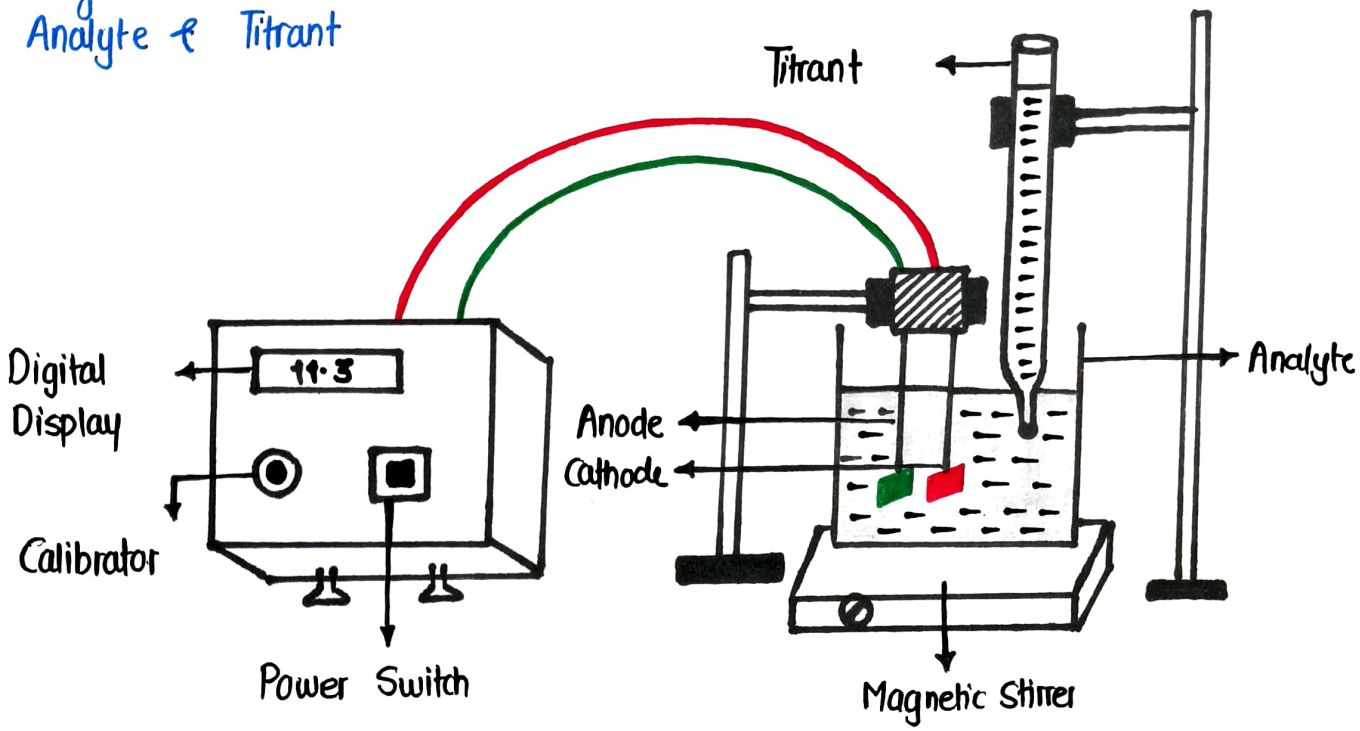
MOLAR CONDUCTIVITY OF SOME IONS

- H^+ : 349.6
- OH^- : 199.1
- Cl^- : 76.35
- NH_4^+ : 73.5
- NO_3^- : 71.46
- Ag^+ : 61.9
- Na^+ : 50.1
- CH_3COO^- : 40.9

Instrumentation Of Conductometry

It consist of a :

- Conductometer
- Conductivity Cell
- Magnetic Stirrer
- Analyte & Titrant



① Conductometer

It consist of a

- Digital Display
- Calibrator
- Power Switch

- Alternative Current (AC) is used as source

② Conductivity Cell

- It is made of Pyrex or Quartz
- It is fitted with two electrodes
  - ⓐ Cathode
  - ⓑ Anode

③ Electrodes

- Electrodes are made of Platinum
- Area = $1\ cm^2$
- Distance b/w Electrodes = 1cm

Procedure

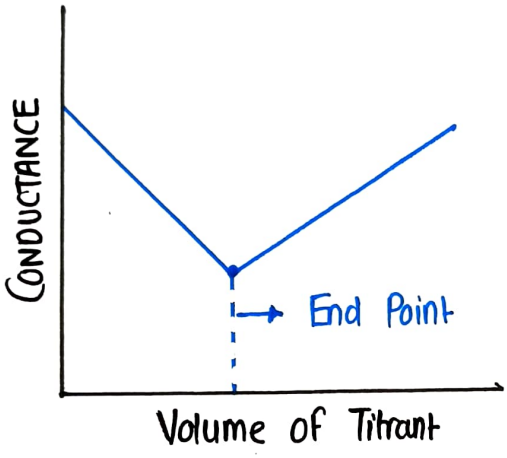
- Prepare the analyte solution & immerse conductivity electrodes.
- Measure the initial conductivity.
- Add titrant in small increments, stirring the solution after each addition.
- Record the conductivity after each addition.
- Plot a graph of conductivity v/s titrant volume
- Identify the equivalence point from the graph.

Application Of Conductometry

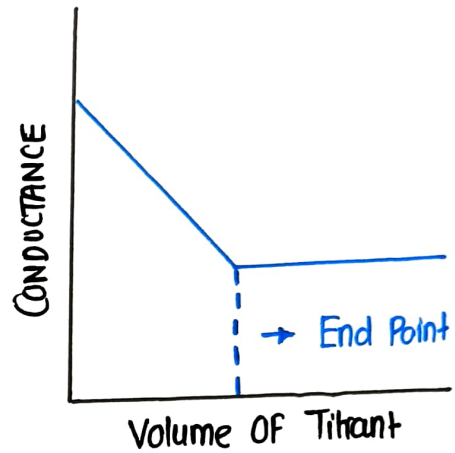
- Check Water Pollution in rivers & lakes
- Determination of Alkalinity of Fresh water
- Determination of Mixture of Acids.

Types Of Conductometric Titration

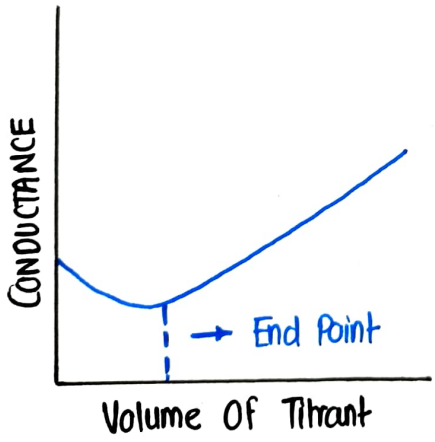
- Strong Acid vs Strong Base
- Strong Acid vs Weak Base
- Weak Acid vs Strong Base
- Weak Acid vs Weak Base



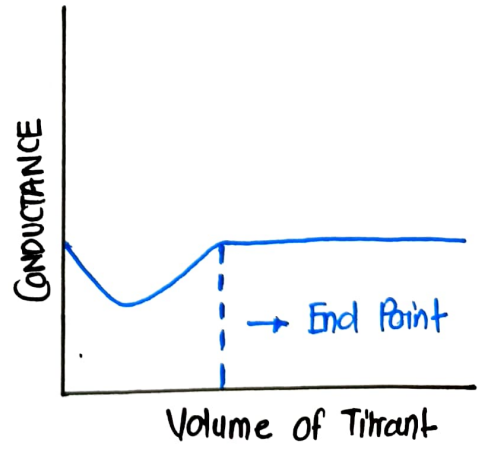
① Strong Acid V/s Strong Base



② Strong Acid V/s Weka Base



Weak Acid V/s Strong Base



Weak Acid V/s Weak Base

POTENTIOMETRY

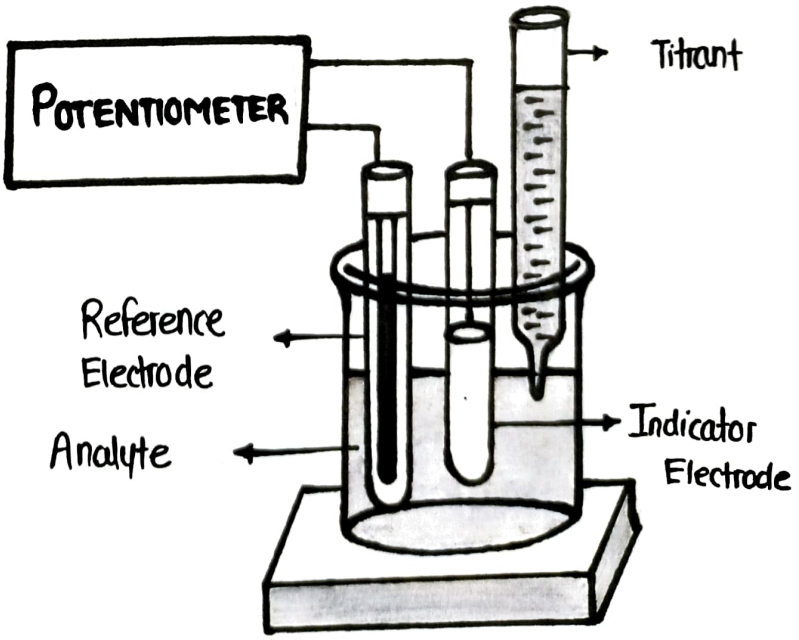
Potentiometry or Potentiometric Titration is an analytical technique used to determine the concentration of an analyte in solution by measuring the change in the electric potential (voltage) of a solution during titration process.

PRINCIPLE

- Potentiometric Titration works by measuring the change in voltage (electric potential) between two electrodes as a chemical reaction takes place during the titration.
- One Electrode (Reference Electrode) keeps a constant voltage, while the other (Indicator Electrode) respond to changes in the concentration of ions in the solution.

INSTRUMENTATION

- Indicator Electrode
- Reference Electrode
- Andyte
- Titrant
- Potentiometer



PROCEDURE

- Prepare the analyte solution and place it into the titration vessel
- Insert the reference and indicator electrodes into the solution.
- Start adding the titrant slowly while stirring.
- Record the potential changes at regular intervals or continuously
- Plot a graph of electrode potential v/s volume of titrant added.
- The end point of the titration is the point at which analyte has completely reacted with the titrant, It is indicated by a sharp change in the potential difference on the titration curve.

REFERENCE ELECTRODES

- A Reference Electrode is an essential component of potentiometric titration that provides a stable and constant potential against which the potential of the indicator electrode can be measured.
- It does not participate in the chemical reaction but serves as a fixed point for accurate voltage comparison throughout the titration process

TYPES OF REFERENCE ELECTRODE

① Standard Hydrogen Electrode
② Saturated Calomel Electrode
③ Silver Chloride Electrode.

① STANDARD HYDROGEN ELECTRODE

It is a reference electrode used in potentiometry to measure the potential change during the titration reaction.

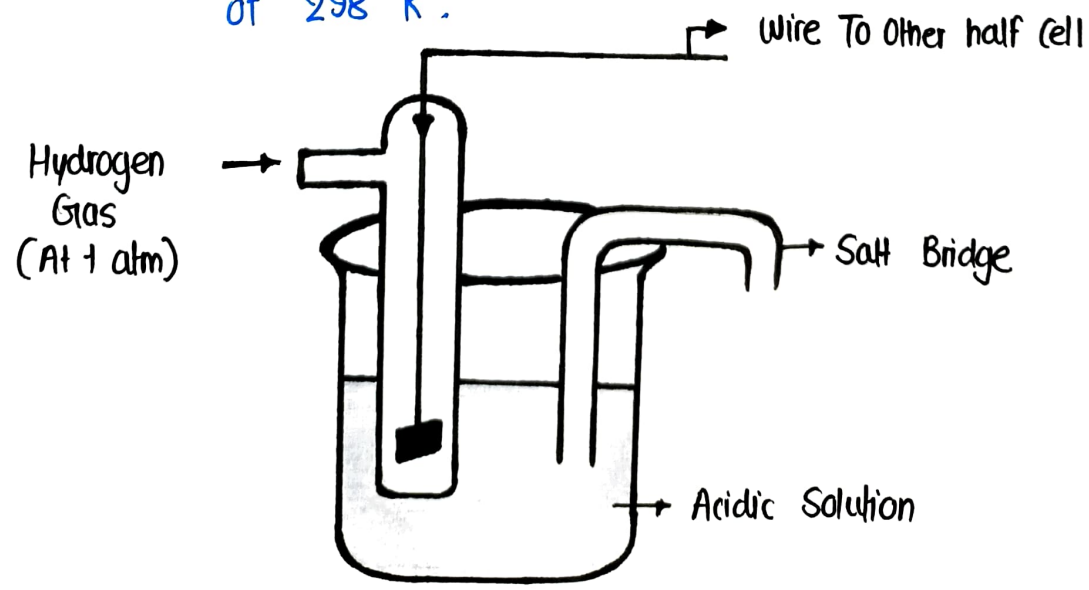
CONSTRUCTION

① Platinum Electrode : • A platinum wire or sheet coated with platinum black is used as electrode.
• Platinum is chemically inert and conducts electron efficiently.

② Hydrogen Gas : Pure hydrogen gas is bubbled over the platinum electrode at a constant pressure of 1 atm.

③ Acidic Solution : The electrode is immersed in an acidic solution, typically containing 1 M hydrogen ions (H^+) from a strong acid like HCl.

④ Temperature : The electrode operates at a standard temperature of 298 K.



APPLICATION

- It is used as primary reference for measuring the electrode potential during titration.
- It is essential in the construction of electrochemical cells.

Advantages

- Provides a stable reference potential
- Can be easily calibrated.

Disadvantages

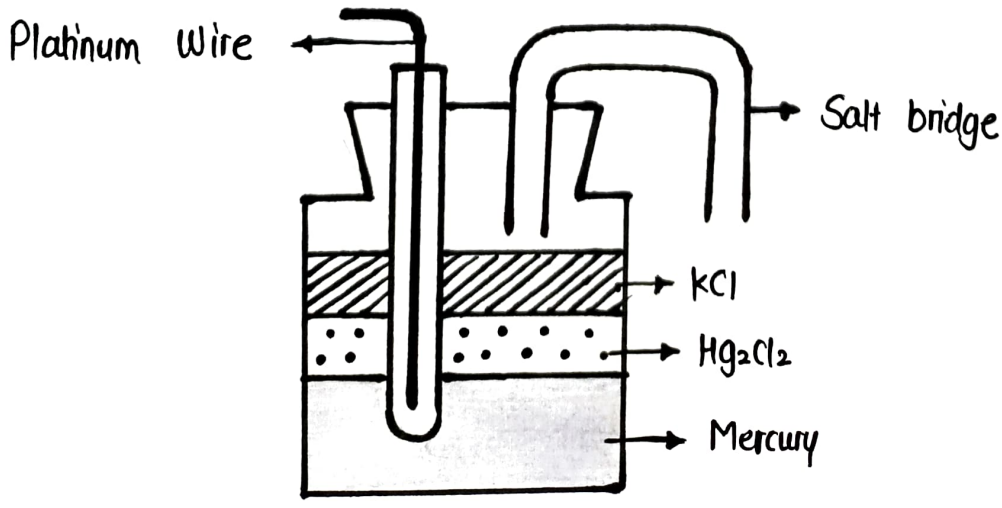
- Handling is risky due to flammability of Hydrogen Gas
- Platinum can be expensive & contaminate.

② SATURATED CALOMEL ELECTRODE

- The saturated calomel electrode is widely used reference electrode in potentiometric titration due to its stable and well defined potential.
- It provides a reliable reference point for measuring the electrode potential of various half cells.

CONSTRUCTION

- Glass Container: A glass vessel holds the components and includes a side arm for contact with external solution.
- Mercury: Pure liquid mercury is placed at the bottom of the container, serving as electrode material.
- Calomel Paste: A layer of mercury chloride (Hg_2Cl_2) is added above mercury.
- Saturated KCl Solution: The container is filled with a saturated potassium chloride solution to maintain constant chloride ion concentration.



APPLICATION

- Electrochemical Studies
- Reference electrode in potentiometry
- pH Measurment
- Corrosion Testing

ADVANTAGES

- Stable Potential
- Easy Maintainance

Limitations

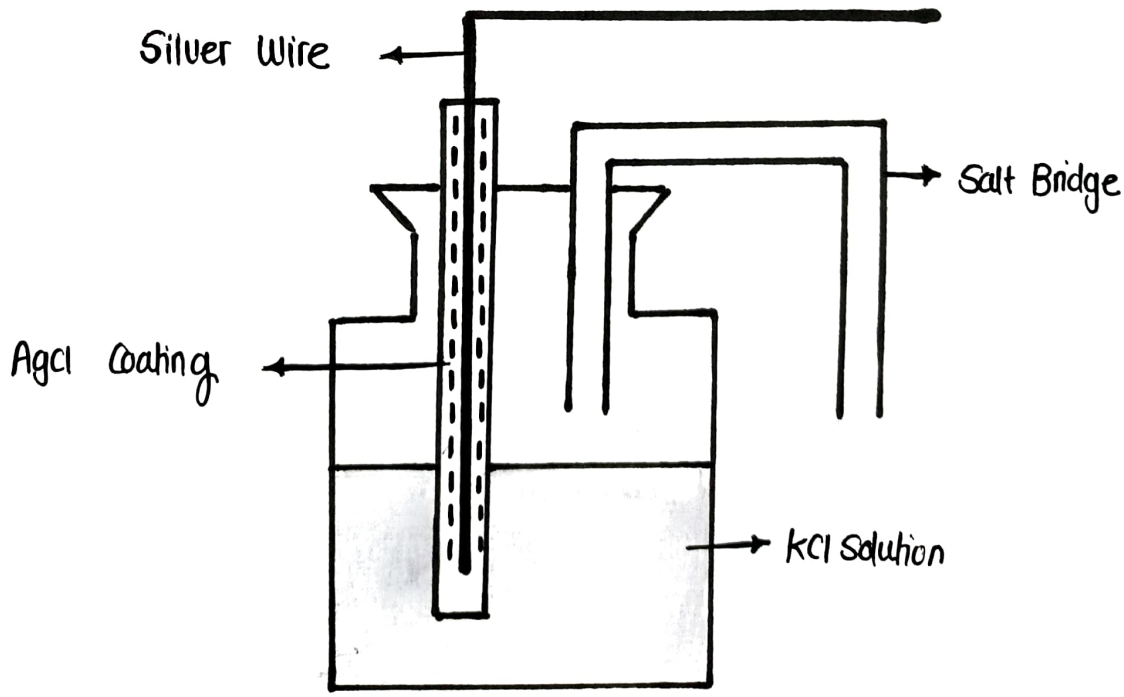
- Mercuric Toxicity
- Temperature Sensitivity

③ SILVER CHLORIDE ELECTRODE

- The Silver - Silver Chloride electrode is a widely used reference electrode used in electrochemical measurments due to its stable and well defined potential.
- It is known for being a safer and more environmental friendly alternative to the saturated Calomel Electrode.

CONSTRUCTION

- Electrode Body: Usually made of glass to hold the components.
- Silver Wire: A pure silver wire is coated with a thin layer of silver chloride through chemical deposition.
- KCl Solution: The electrode is immersed in a fixed concentration of KCl solution.



APPLICATION

- pH Measurment
- Potentiometric Titration
- Corrosion Monitoring

ADVANTAGES

- Stable Potential
- Environmentally Safer
- Temperature Compatability

DISADVANTAGES

- Electrolyte Leakage
- Chloride Ion Dependence

POLAROGRAPHY

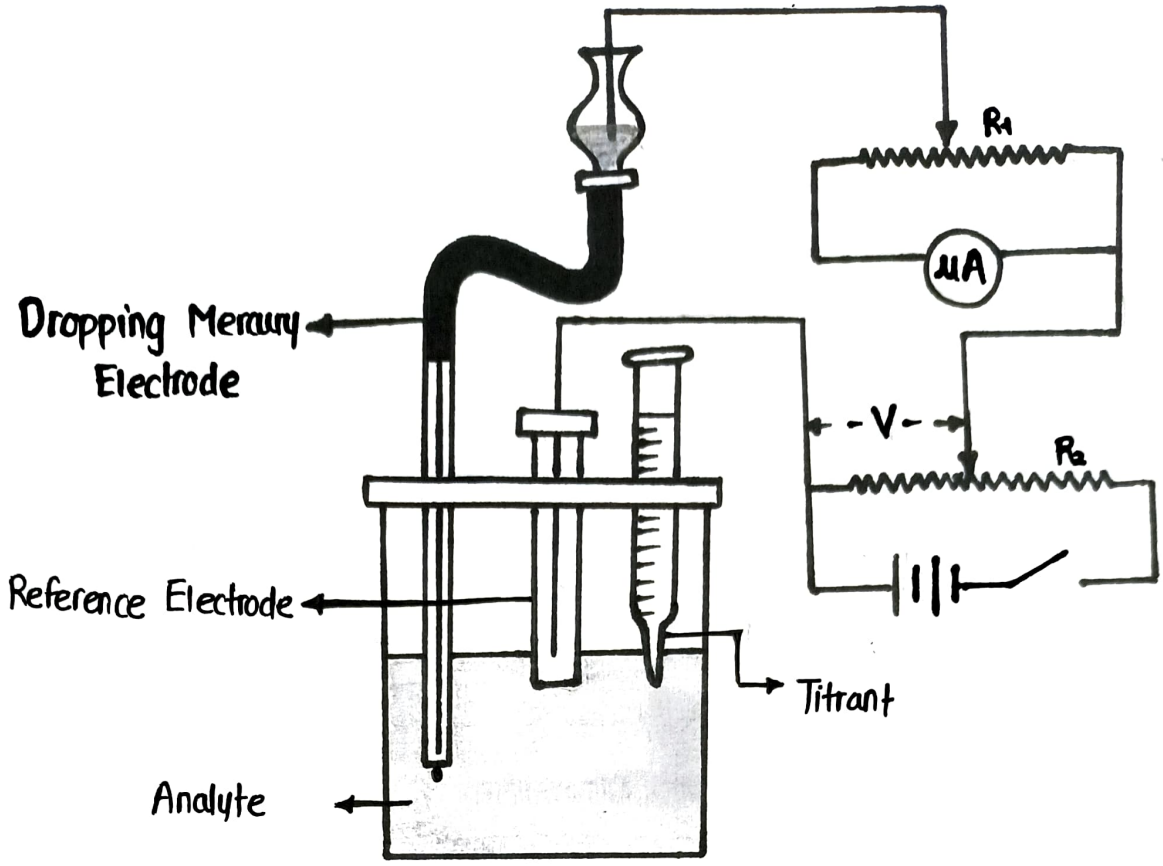
Polarography is an electrochemical analytical technique that measures the current flowing through a solution as a function of an applied voltage, typically using a dropping mercury electrode as the working electrode.

PRINCIPLE

- In polarography a linearly increasing voltage is applied between the working electrode and a reference electrode.
- When the applied potential reaches the point where a chemical in the solution starts to gain or lose electrons, a noticeble current change occurs.

COMPONENTS

- Dropping Mercury Electrode (DME) : Consist of a glass capillary through which mercury flows, froming droplets at its tip.
- Reference Electrode : Commonly a saturated calomel electrode.
- Analyte : Solution to be analysed.
- Polarograph: Instrument that applies voltage and measures current.



PROCEDURE

- Prepare the analyte solution.
- Setup the polarographic cell with the electrodes properly immersed.
- Apply a linearly varying voltage across working & reference electrode
- Record the current.
- Measure the concentration using Ilkovic equation

$$id = 607 \times n \times D^{1/2} \times m^{2/3} \times t^{1/6} \times C$$

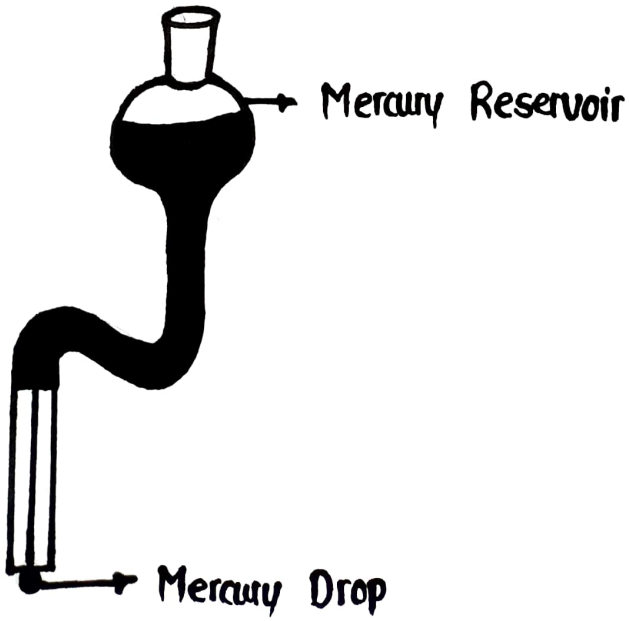
- Here id = Diffusion current
  n = No of electrons involved in redox reaction
  D = Diffusion Coefficient
  m = Rate of mercury drop from the electrode
  C = Concentration of analyte
  t = Drop time of mercury

DROPPING MERCURY ELECTRODE

- A Dropping Mercury Electrode is an electrode commonly used in polarography.
- It consist of a thin capillary tube from which mercury drops continuously and slowly fall into the solution under gravity.
- Each drop provides a fresh, clean and smooth surface for electrochemical reactions.

WORKING

- The DME functions by continuously forming mercury drops at the tip of a fine capillary tube under the influence of gravity.
- These drops fall at regular intervals, typically every few seconds, providing a clean, renewable electrode surface.
- A linearly increasing potential is applied b/w the DME and reference electrode.
- As voltage increase, analyte undergoes oxidation or reduction (electron transfer)
- This reaction generates an electric current, which is then recorded for identifying the concentration of analyte



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