# تفريغ لاب تحليل آلي

اسم الموضوع: Potentiometeric Titration

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Seul Academy

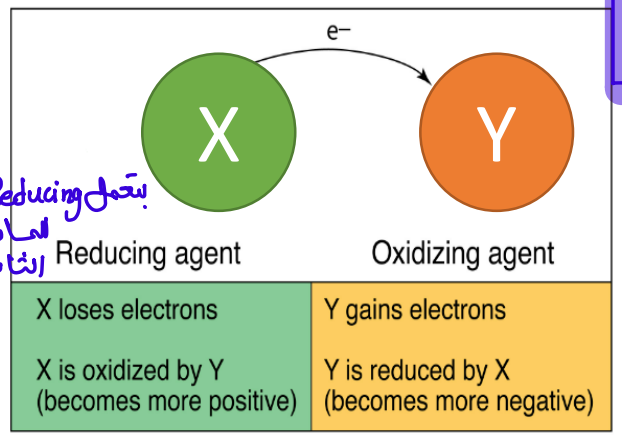
لجان الدُّفعات

# Experiment 2

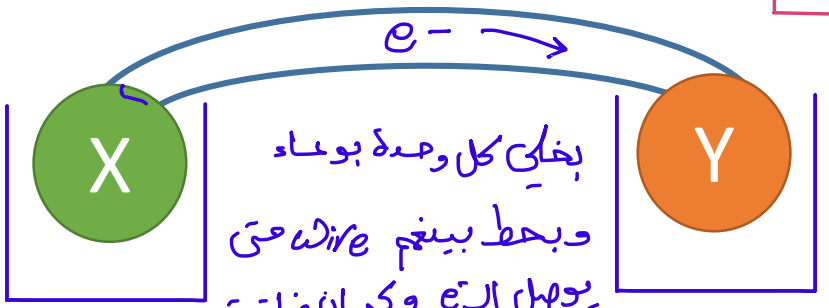
# Potentiometric Titration (pH meter)

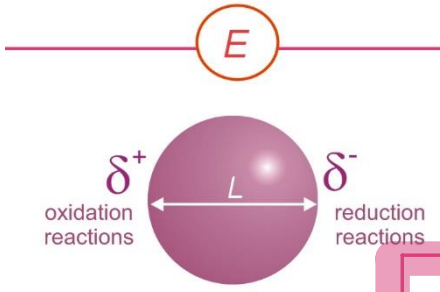
# Definitions:

- **Electrochemistry: Conversion of electrical energy (electricity) to chemical energy (chemical reaction) or vice versa.**




Chemical → electrical

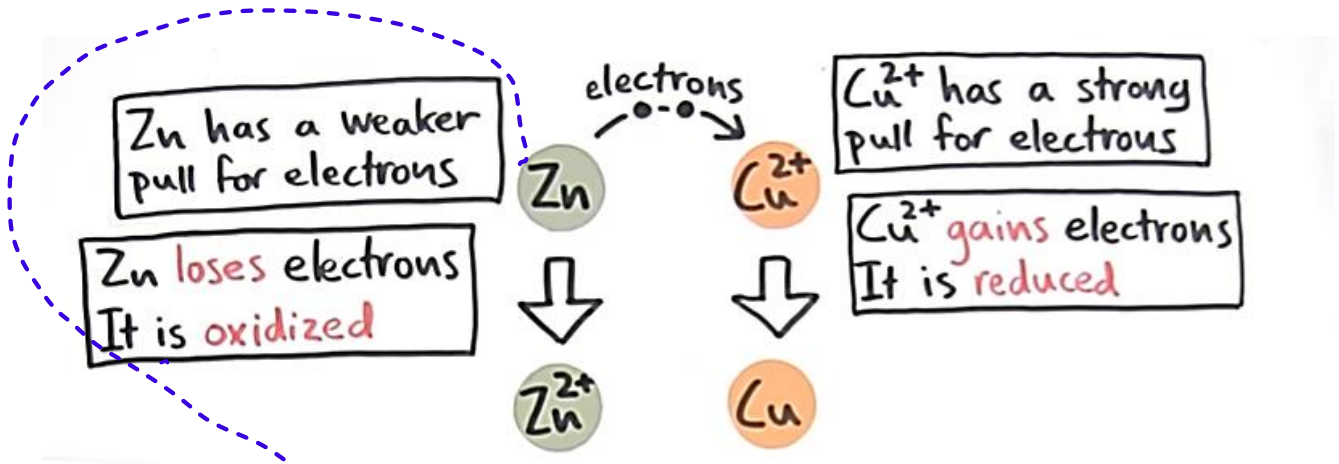


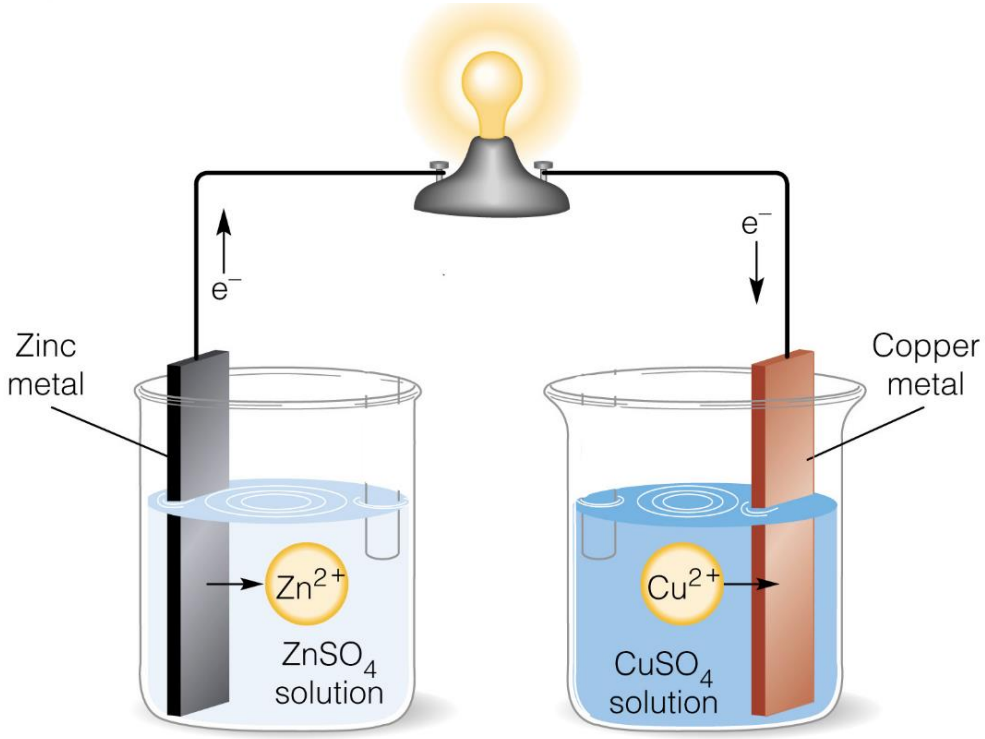

eletrical → Chemical

مثال عملي

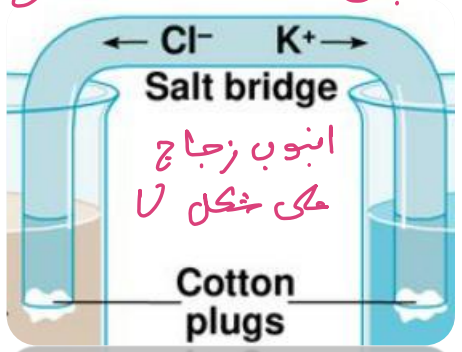
# How can we utilize the chemical reaction to make electricity?




من ناحية
Potential energy
عنده tendency انه يخسر $e^-$
اكثر من Cu
لكهربائية
oxidation



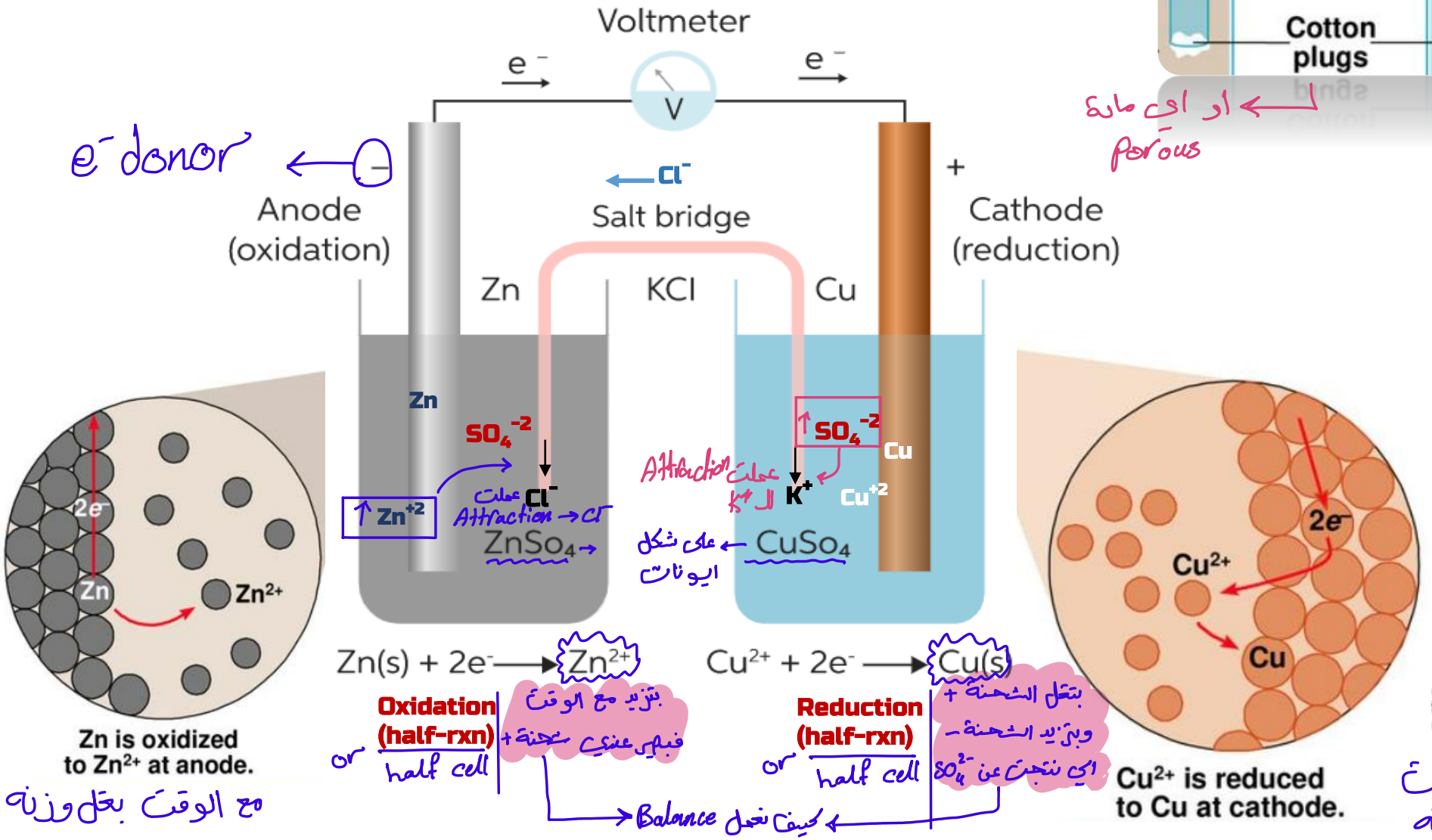
# Galvanic Cell




عبارة عن محلول ملح

انبوب زجاج على شكل U

او اي مادة Porous




عملت Attraction → Cl⁻

عملت Attraction ← K⁺ ال

على شكل ← ايونات

بتزيد مع الوقت فبيعطي عنصي شحنة +

بتقل الشحنة + وبتزيد الشحنة - اي نتجت عن $SO_4^{2-}$

or

or

كيف نعمل

مع الوقت بتقل وزنه

مع الوقت بزيد وزنه

بين ال half cell ؟
Salt bridge

# Potentiometric titration

معتمدة على ← يعني على فرق Potential
الجهد الكهربائي

الفرق عن ال titration العادي

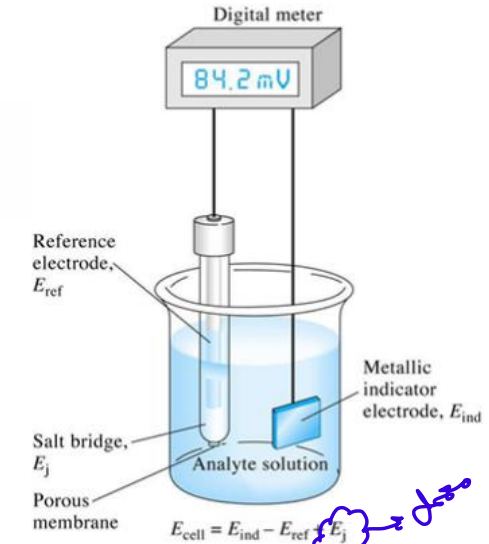
**A technique whereby the chemical reaction is converted to electrical energy. No indicator is used, instead it uses a *potentiometer* (ex: pH meter) to measure the amount of an analyte present in the given solution by measuring the change in the potential difference between two electrodes (working & reference electrodes) immersed in the analyte as a function of successive addition (volume) of titrant of known concentration till the endpoint.**

مغمور

متتالية

- **Potentiometric titrations can be divided into four categories based on the type of reaction taking place:**
  1. **Complexometric titration** complexation Rxn
  2. **Precipitation titration** ppt بيتكون منه
  3. **Redox titration** oxidation - Reduction Rxn
  4. **Acid-Base titration**

✨💕 تجربة اليوم



A cell for potentiometric determinations

محمل

❑ **Potentiometric cell is composed of the following:**

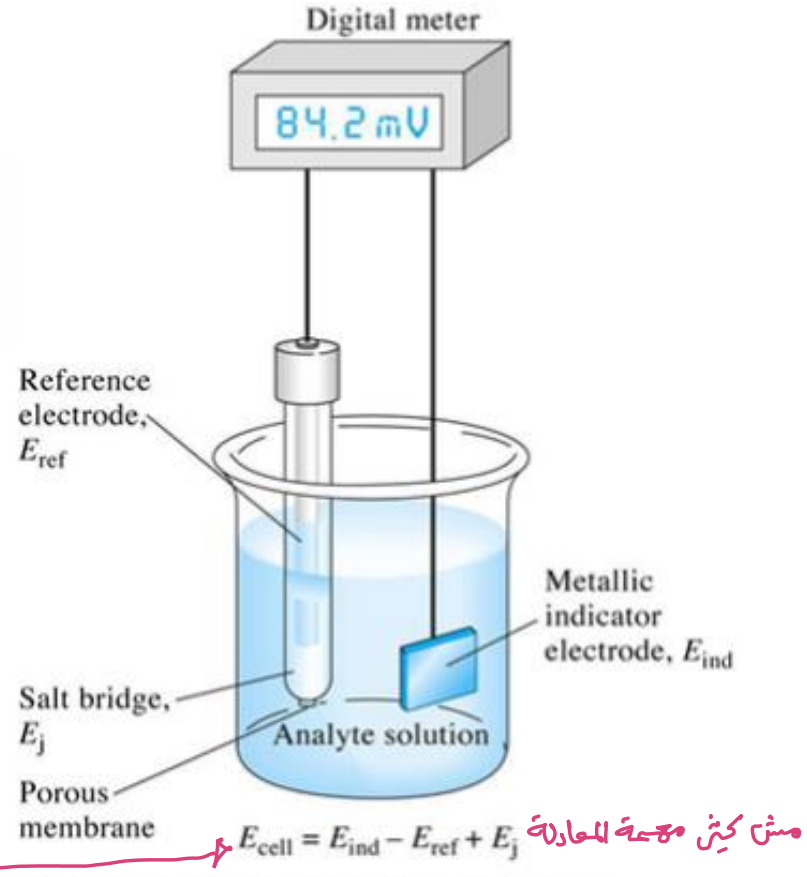
**1) Two Electrodes:**

- **Standard Reference Electrode**
- **Working/Indicator Electrode (Sample)** ⟹ هو اللي بيتأثر في بالسامبل

**2) Salt bridge : Junction** ↝ رابط

**3) Wire**

**4) Voltmeter: measures ΔE (potential difference) developed by the cell, between the indicator electrode and the reference electrode.**



مش كتير معجبة المعادلة

A cell for potentiometric determinations

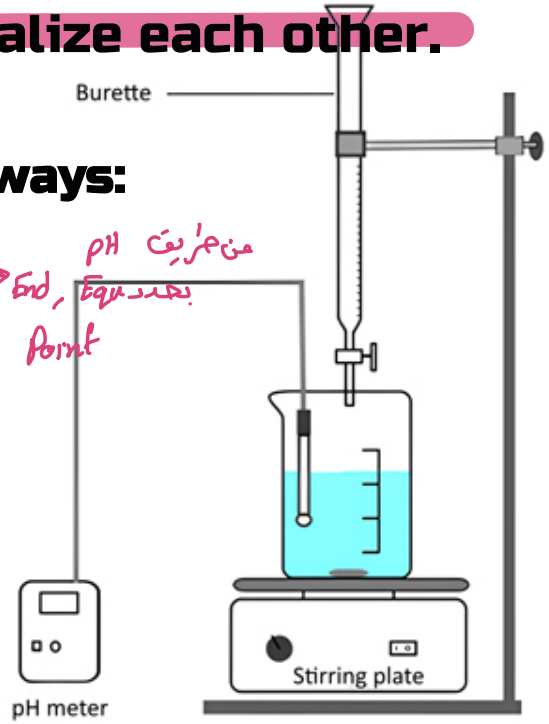
❑ **Potentiometry is governed by the Nernst Equation**

بعتمد الجهد الخلية

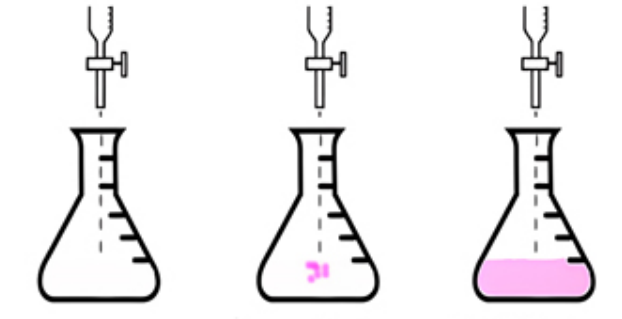
# Acid-Base Titration (Neutralization Titration)

**This type of titration is used in potentiometry to calculate the concentration of an acid/ base using another base/acid with a known concentration, respectively. An acid and a base neutralize each other.**

- **This titration can be performed in two ways:**
  - **A. Using a potentiometer (pH meter).**
  - **B. Using a color indicator.**




A




B

## ❖ Standard reference electrode:

**is a half cell with an accurately known constant electrode potential, that is independent on (completely insensitive) the concentration of the analyte or any other ions in the solute under study (ex: Calomel Electrodes and Silver-Silver Chloride Electrodes).**

## ❖ Indicator electrode:

**is a half cell that develops a potential, that depends on the activity of the analyte, and it responds rapidly and reproducibly to changes in activity of the analyte ion (ex: metallic and membrane (Ion Selective Electrodes : ISE → *glass electrode*).**

## ❖ Salt bridge:

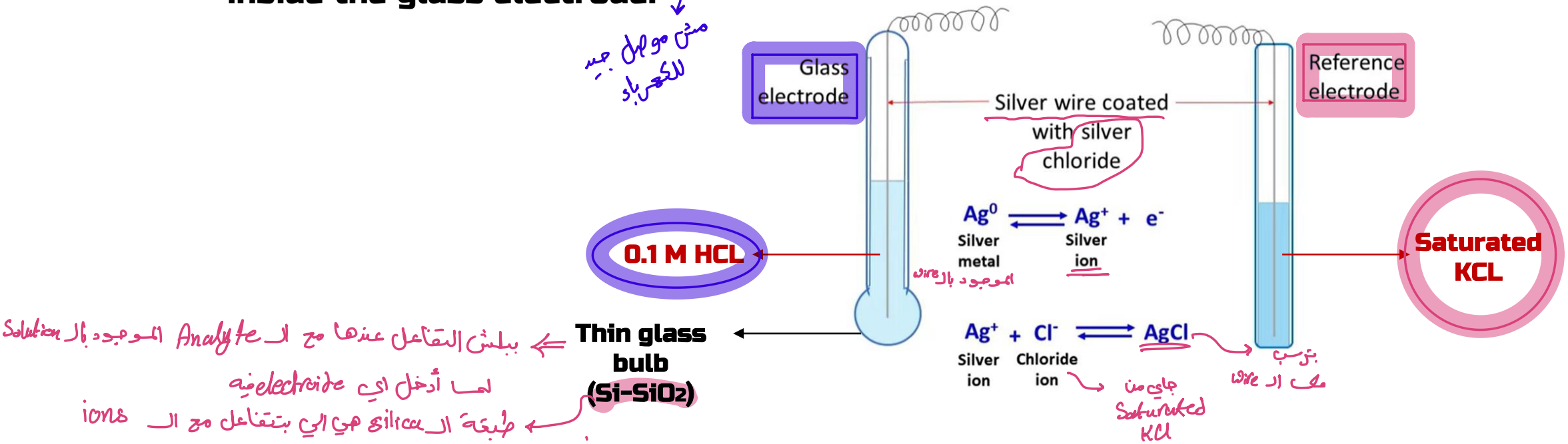
**Prevents the components of the analyte solution from mixing with those of the reference electrode. Potassium chloride (KCl) is a nearly ideal electrolyte for the salt bridge because the mobilities of the $K^+$ ion and $Cl^-$ ion are nearly equal. Thus, canceling the potential that develops at each end of the bridge.**

# pH meters

**Like any electrochemical cell, pH meter consists of two electrodes:**

- **Reference electrode → Ag-AgCl electrode**
  **A silver wire coated with a layer of silver chloride that is immersed in KCl solution saturated with AgCl)**
  **Ag | AgCl(sat'd), KCl**

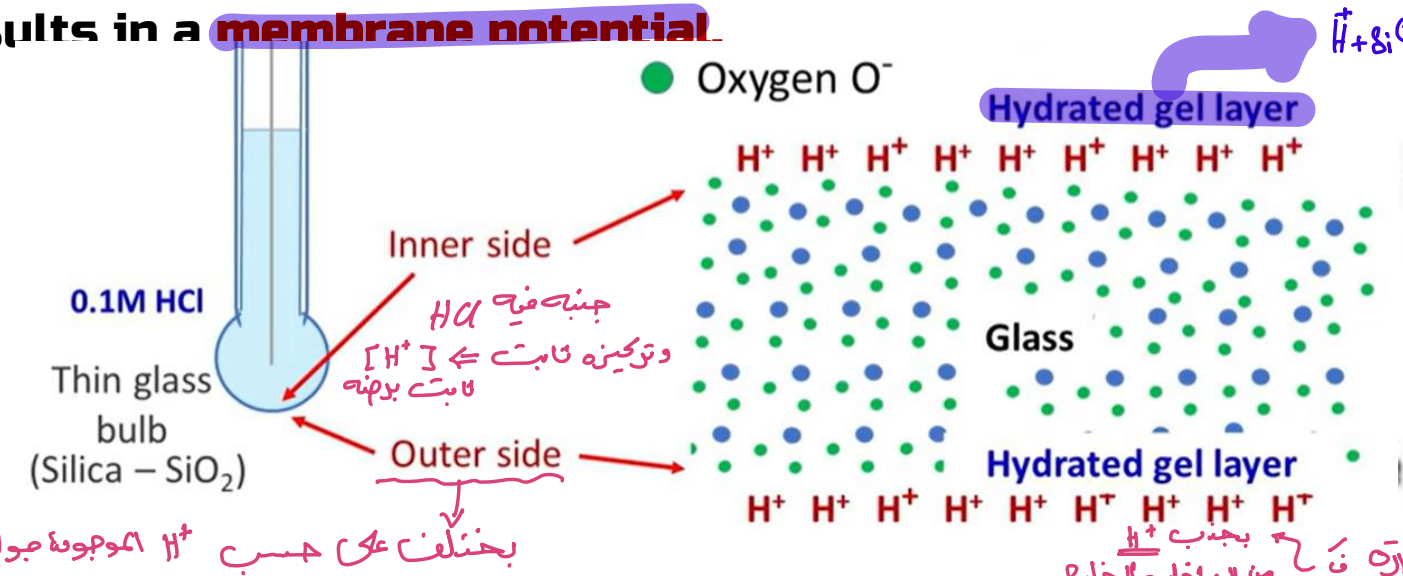
- **Indicator electrode → Glass electrode which is made of (Silica-$SiO_2$)**
  **Since glass is not a conductor, an internal reference electrode (Ag-AgCl) is placed inside the glass electrode.**

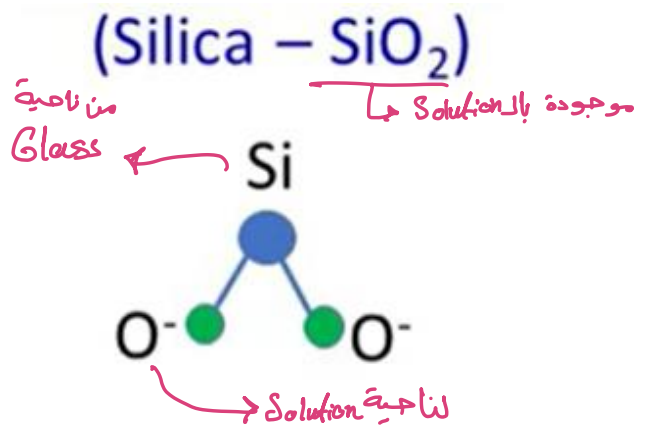


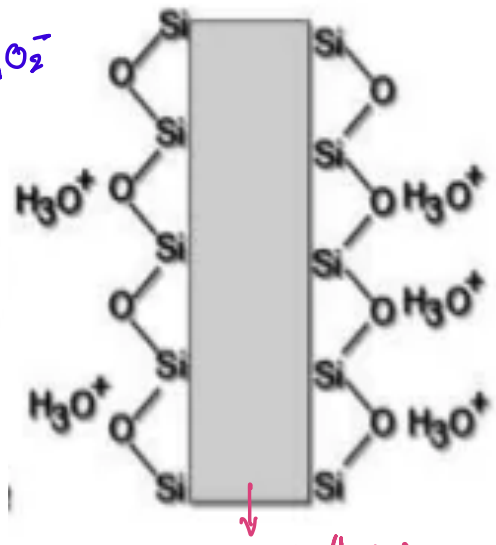
- **Both the inside and outside surfaces of the glass membrane in the GE bulb have SiOH groups. The interior surface of the glass membrane is in contact with a constant concentration of HCl, and so the number of $SiO^-$ groups on the interior surface remains constant.**

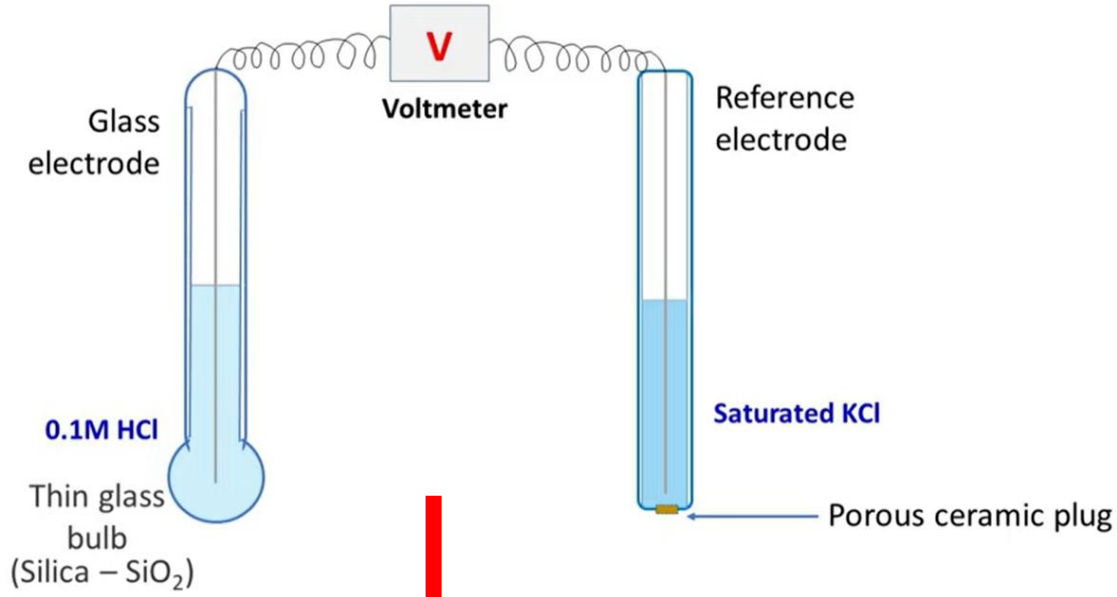
- **By contrast, the number of $SiO^-$ groups on the exterior of the glass membrane will change when the pH of the solution, in which the glass membrane is immersed, changes.**

- **The difference in charge on the inside and outside of the glass membrane results in a membrane potential.**

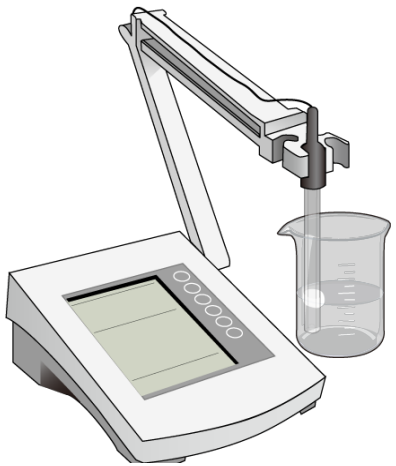


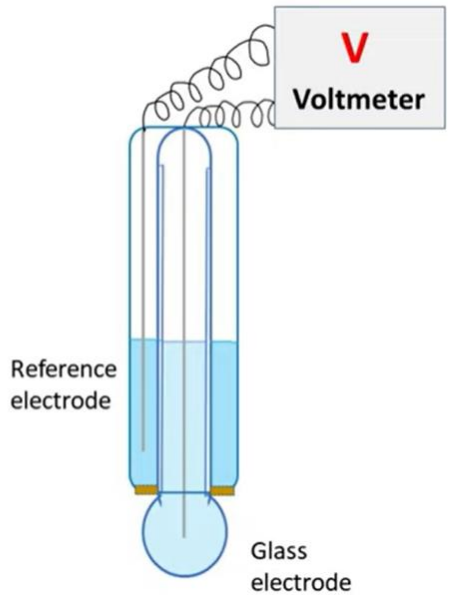



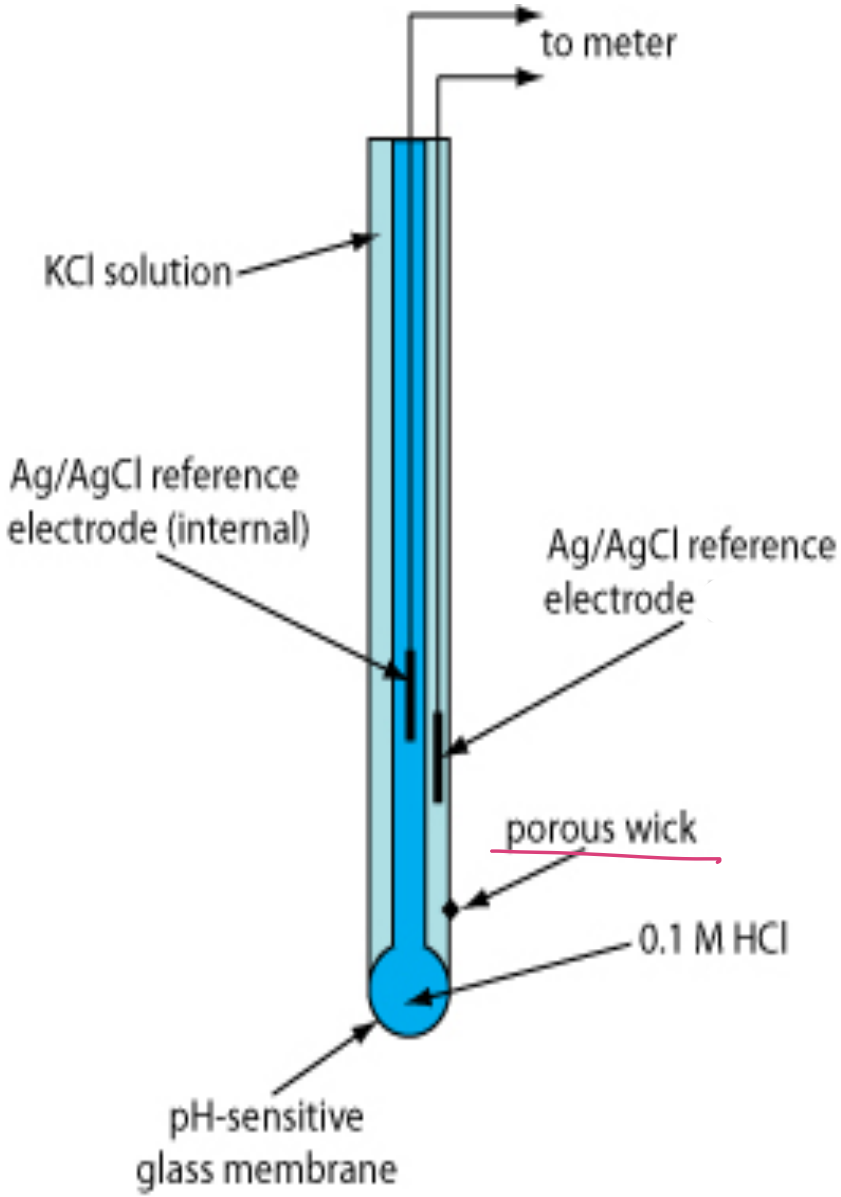






Combined electrode

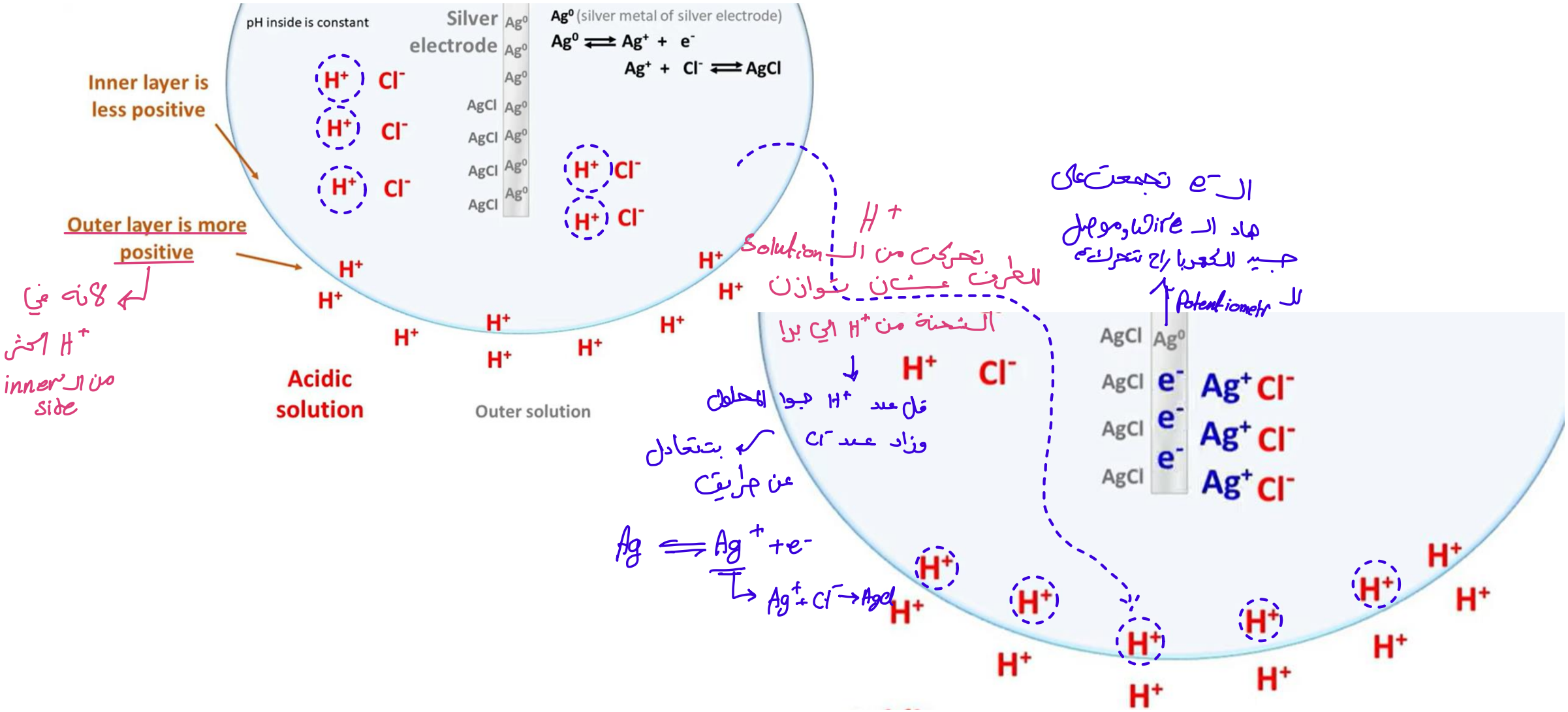




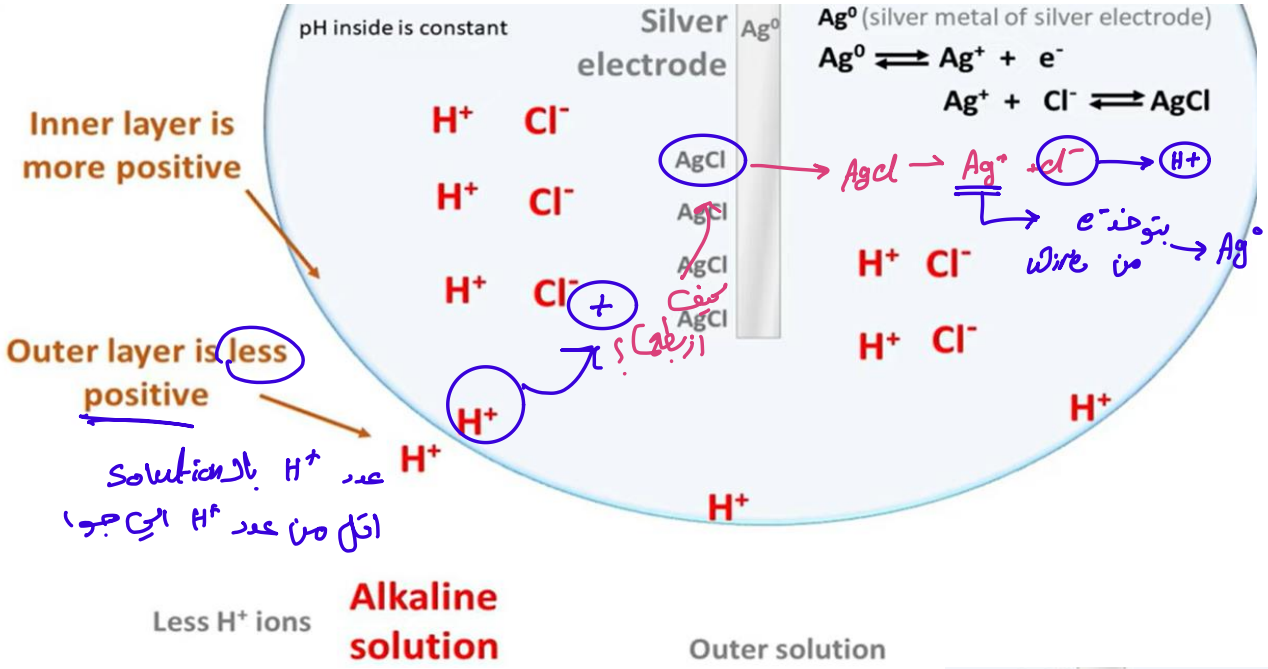



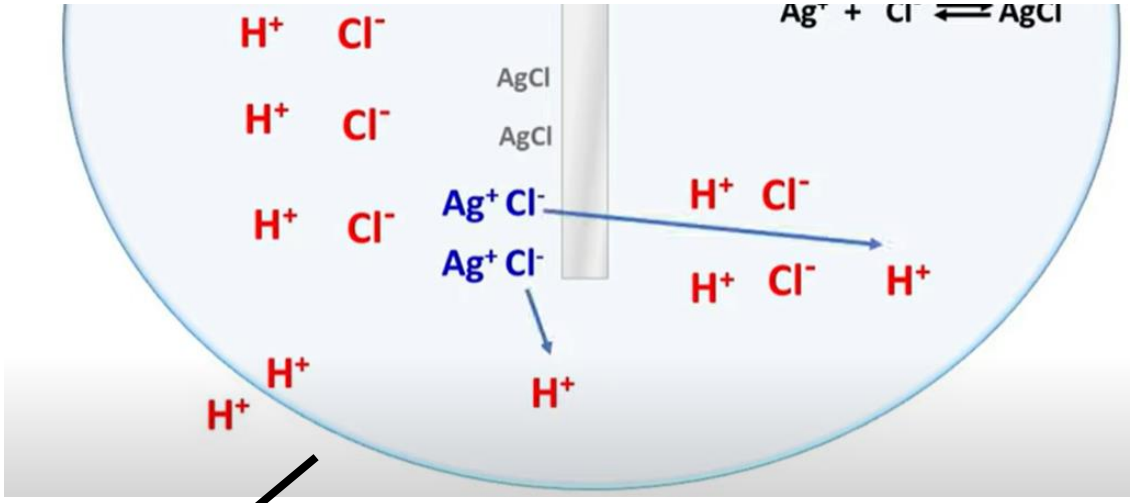
# Acidic Solution → High concentration of H+ in the sample

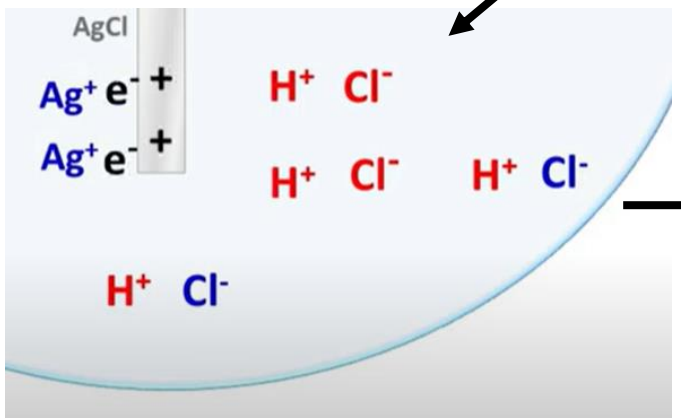
↑ [H+]

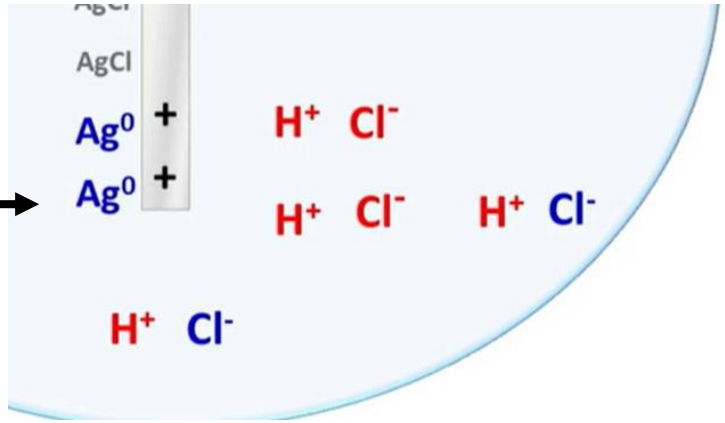


# Alkaline Solution → Low concentration of H+ in the sample

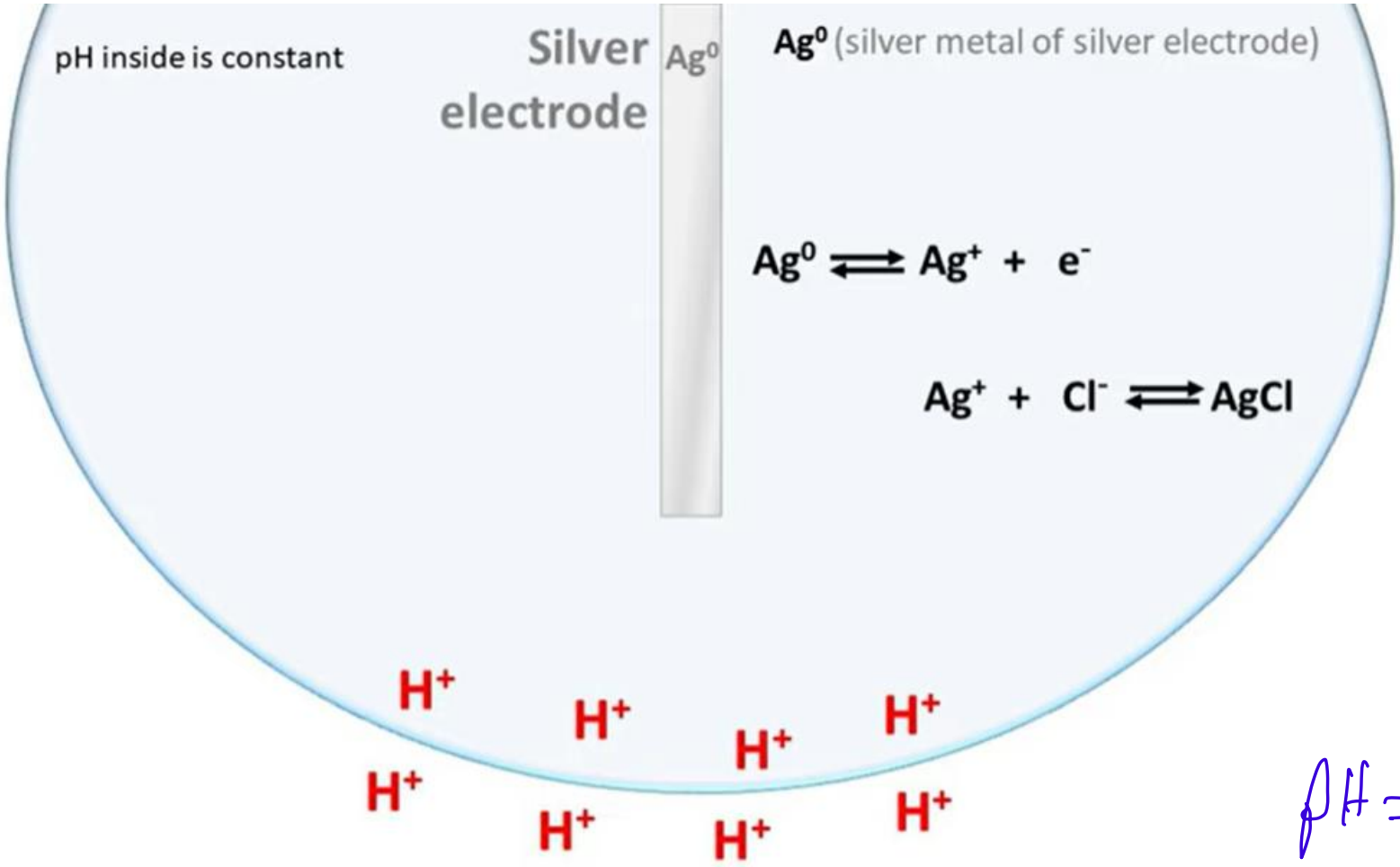


# Neutral Solution

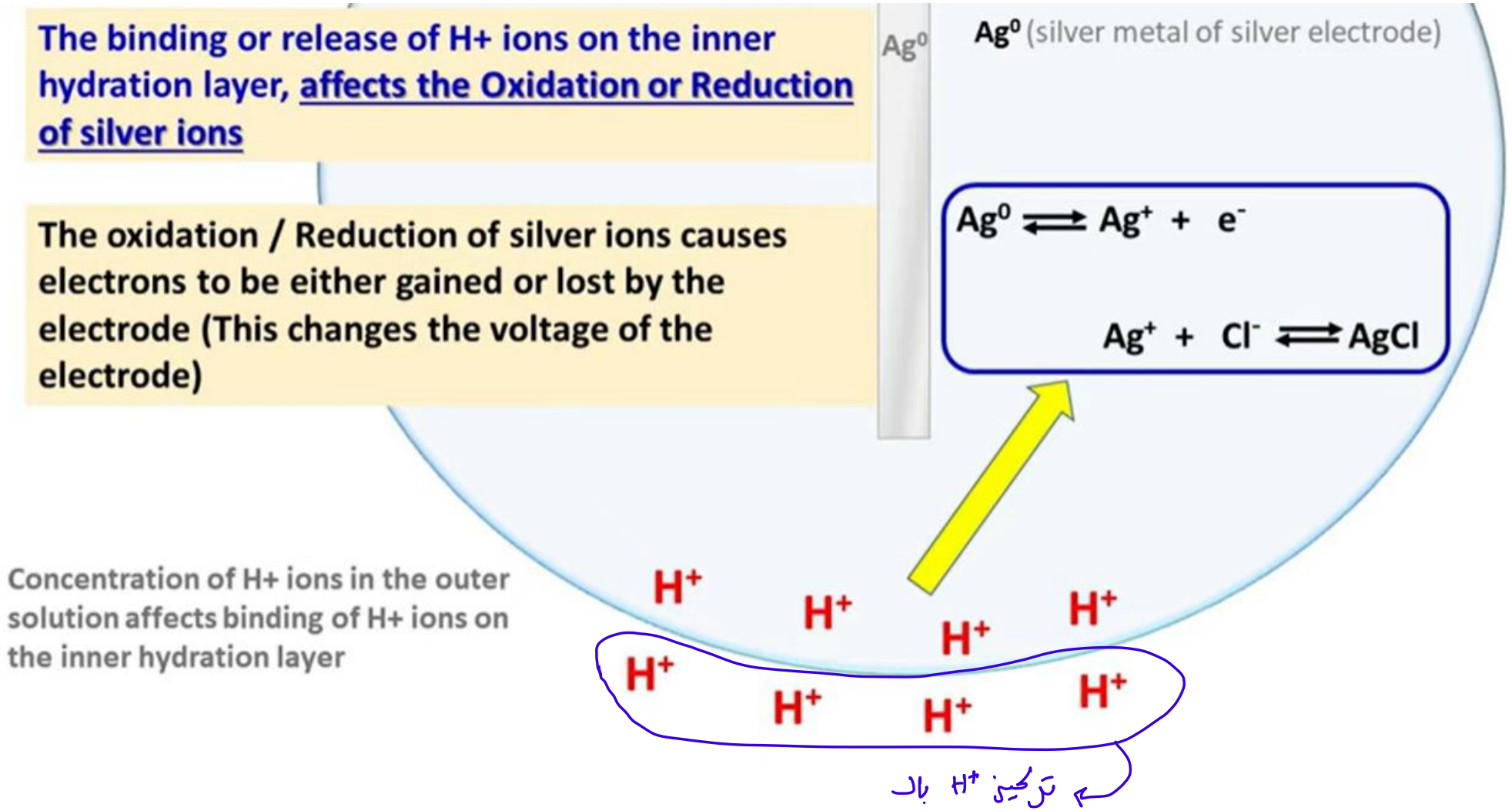



pH = 7

# Summary

The binding or release of H+ ions on the inner hydration layer, affects the Oxidation or Reduction of silver ions

The oxidation / Reduction of silver ions causes electrons to be either gained or lost by the electrode (This changes the voltage of the electrode)

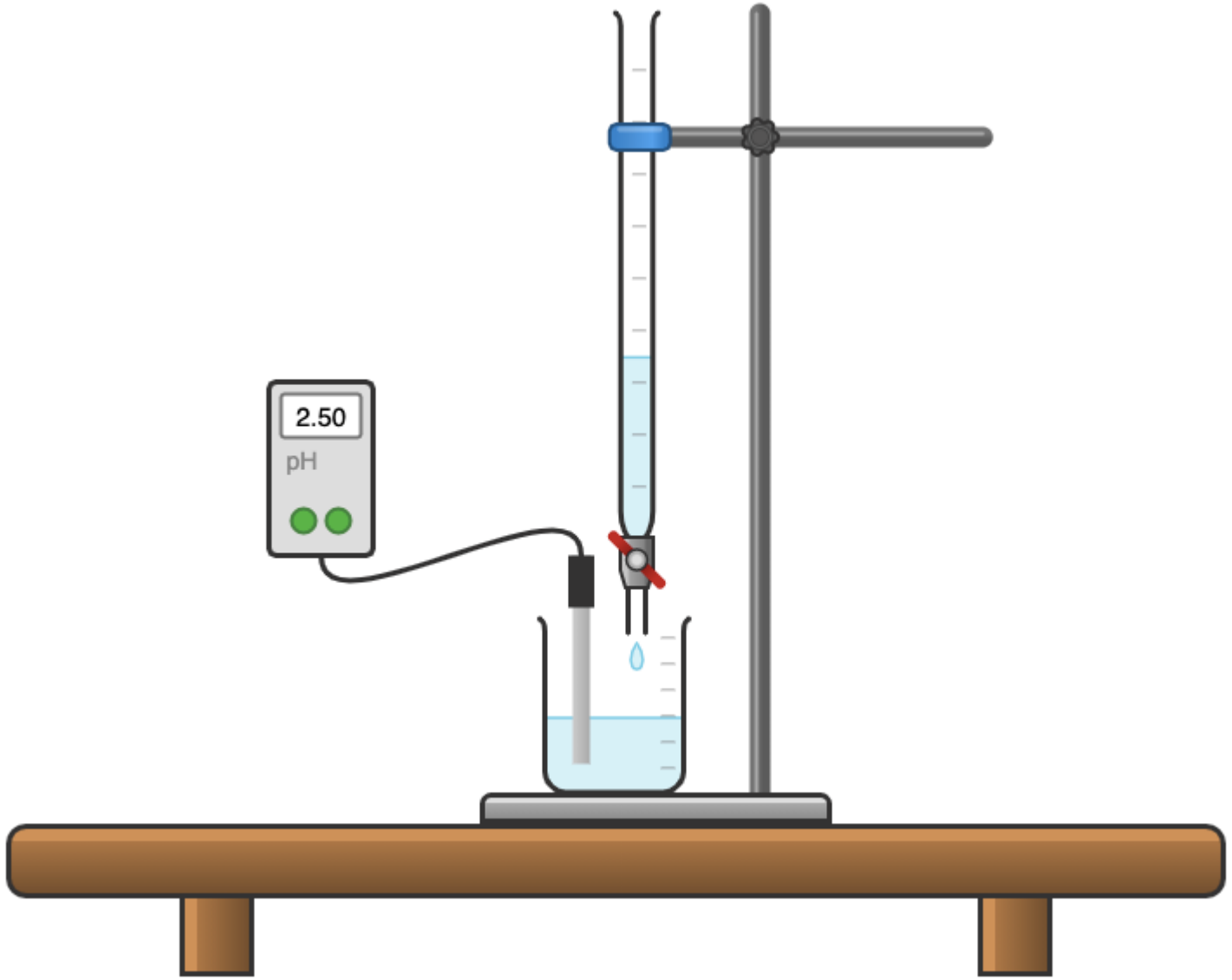



Concentration of H+ ions in the outer solution affects binding of H+ ions on the inner hydration layer

تركيز $H^+$ بال Outer solution بأثر بشكل مباشر على $H^+$ المرتبطة على inner Hydrating Gel

# pH titration of a weak acid against a strong base can be used for:

- **Qualitative Analysis** → **determine the type of weak acid  by its pKa**
- **Quantitative Analysis** → **determine the concentration of the weak aid**



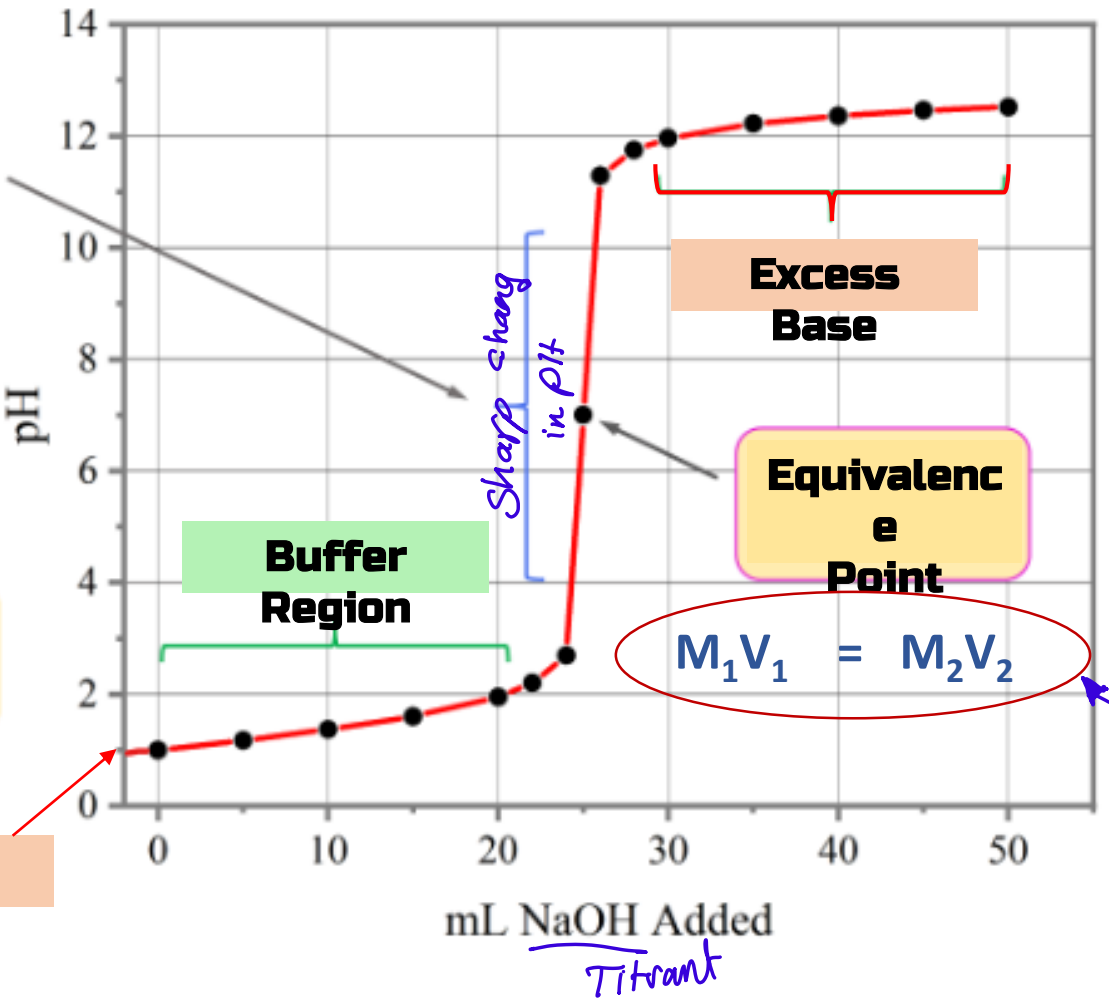
# Four parts of titration curve

*Near the equivalence point, the concentration of the $H^+$ and $OH^-$ is almost equal and that is why the addition of even a single drop of titrant causes an abrupt change in the pH.*

**Before equivalence** point ($H^+$ in excess)

pH < 7

**Pure Acid**




**After equivalence** point ($OH^-$ in excess)

pH > 7

$HA \rightleftharpoons H^+ + A^-$

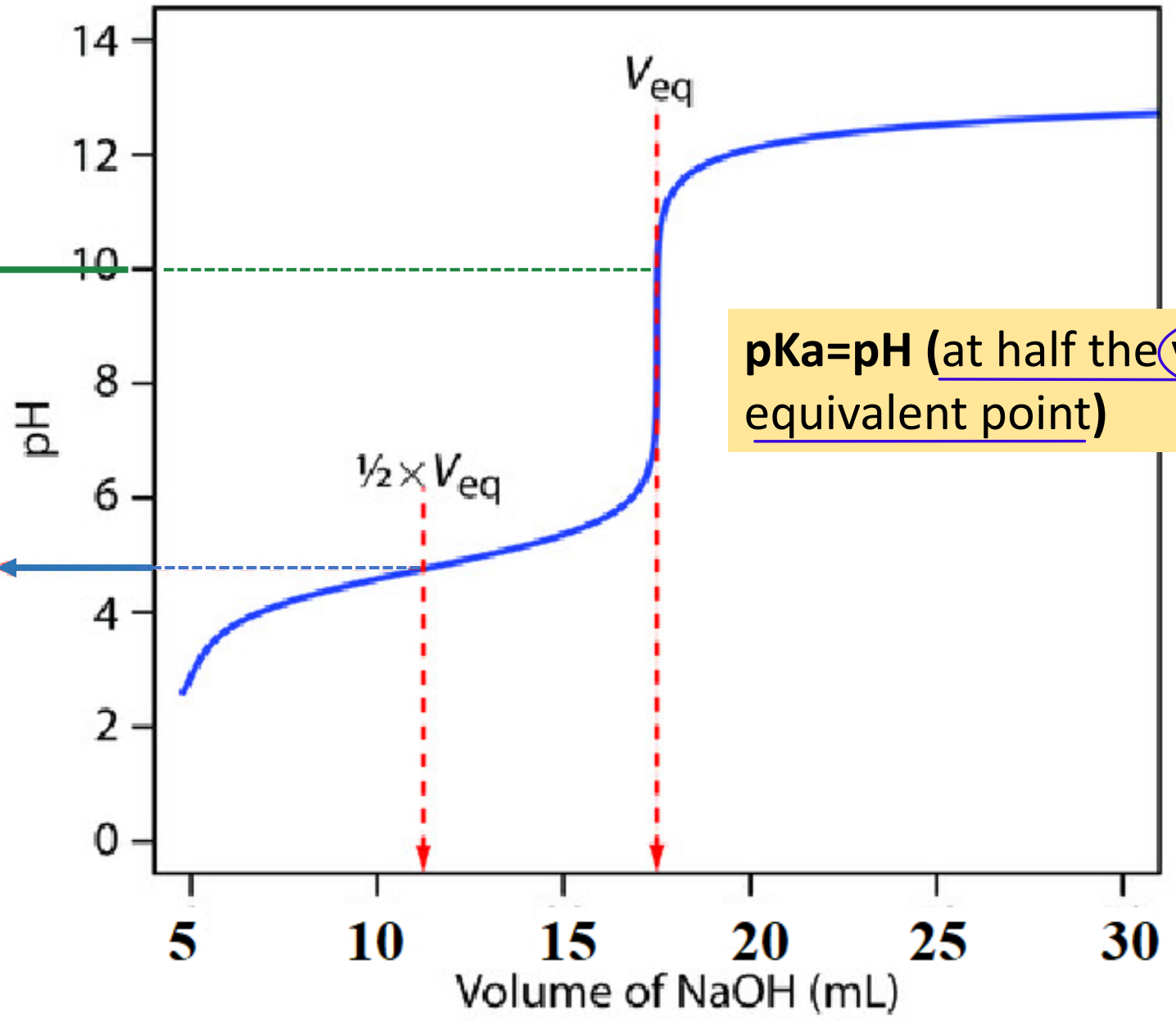
$[H^+] = [OH^-] = 10^{-7}\ M$

pH = 7

*At the equivalence point only NaCl, equal amounts of $[H^+]$ and $[OH^-]$, from the autoionization of water, are present in the solution.*

**pH**
**of the Eq. point**

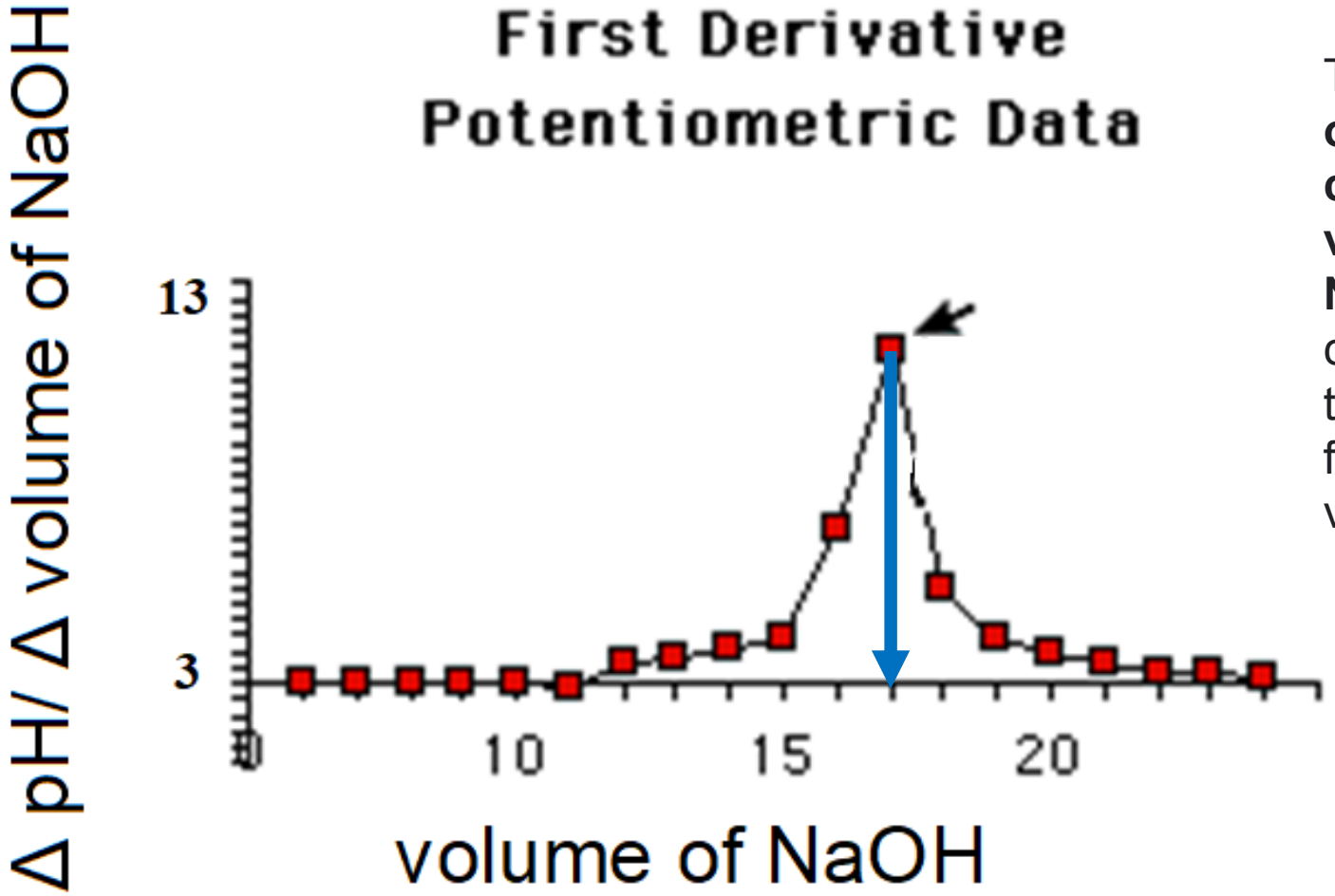
**pKa = pH**




**pKa=pH (**at half the volume of the equivalent point**)**

Titrant

**At the volume (V') where (ΔpH/ΔV) has the maximum value in a first derivative plot. (V') is the average volume of two subsequent volumes.**

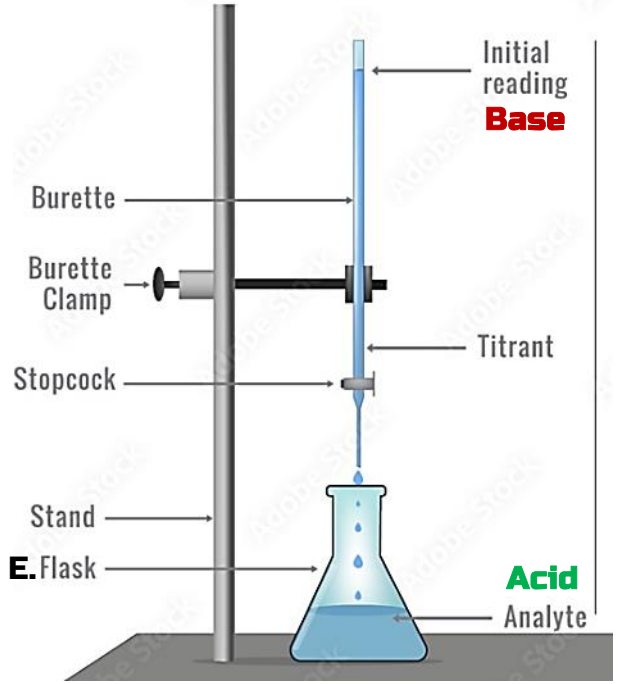



This **plots the change of pH divided by the change in volume versus the volume of NaOH**. This shows the change in slope of the titration curve as a function of the added volume of base.


# Practical Part

# Glassware & Materials

**Each group will be supplied by the following glassware:**



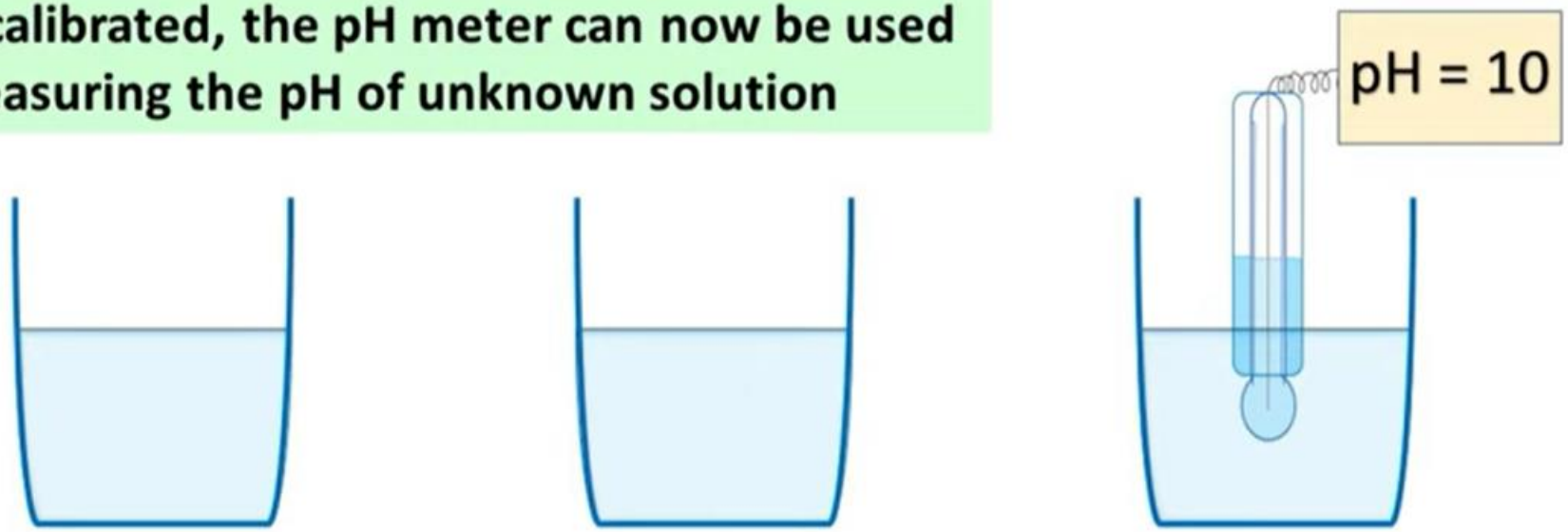

| GLASSWARE | CHEMICALS |
|---|---|
| 10 ml volumetric pipette | Unknown acid solution |
| Graduated Cylinder 50ml | Distilled water |
| 250 ml Erlenmeyer flask or Beaker | 0.1 M NaOH |
| Burette | |
| Stand for titration | |

# Instrument

# Calibration of the pH meter

Practically, For the accurate measurement of pH, the pH meter must be calibrated

Once calibrated, the pH meter can now be used for measuring the pH of unknown solution




pH = 4 Buffer

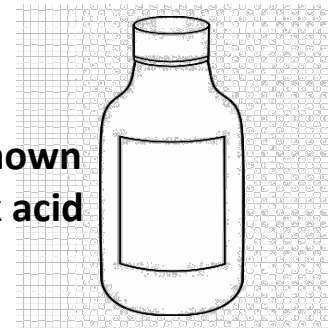
pH = 7 Buffer

pH = 10 Buffer

For this the buffers with known pH are used

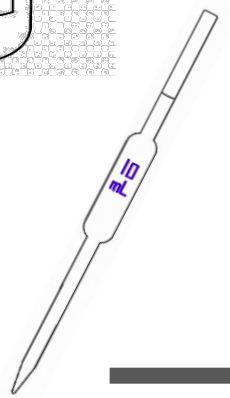
# Procedure

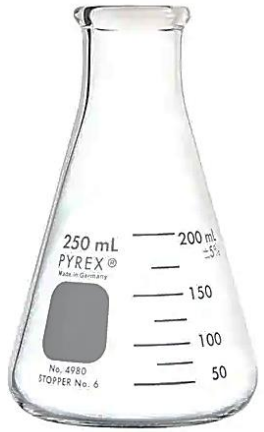
1. Pipette 10ml of your unknown acid sample into a clean conical flask.
2. Add 50ml distilled water to the sample.
3. Measure the pH of the solution in the conical flask before starting titration.
4. Titrate the above solution with 0.1M NaOH solution in an increment of 2ml and record the pH value after each addition in a draft paper.
5. Continue the titration until the PH is greater than 12.5
6. Determine the end point roughly.
7. Titrate the second sample solution by adding 1ml of the titrant gradually until the beginning of the potential jump, determined from the first trial is reached, then continue adding NaOH in 0.2ml increment until the end of the potential jump determined from the first trial is reached.
8. Continue the titration in 1ml increment addition until the PH is greater than 12.5
9. Record the no. of mls of NaOH and the PH of the solution after each addition in the report sheet.



Unknown weak acid

Place 10 ml of the Unknown in a 250ml flask



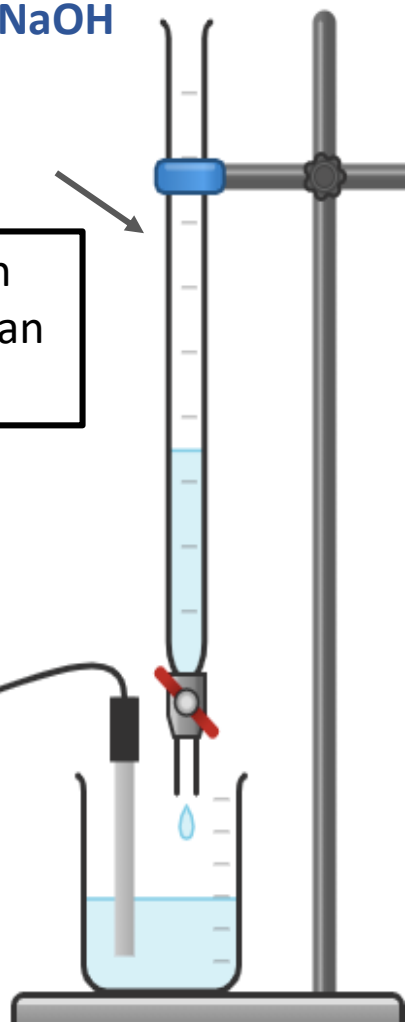



Add 50 ml distilled water

Fill the burette with 0.1 M NaOH

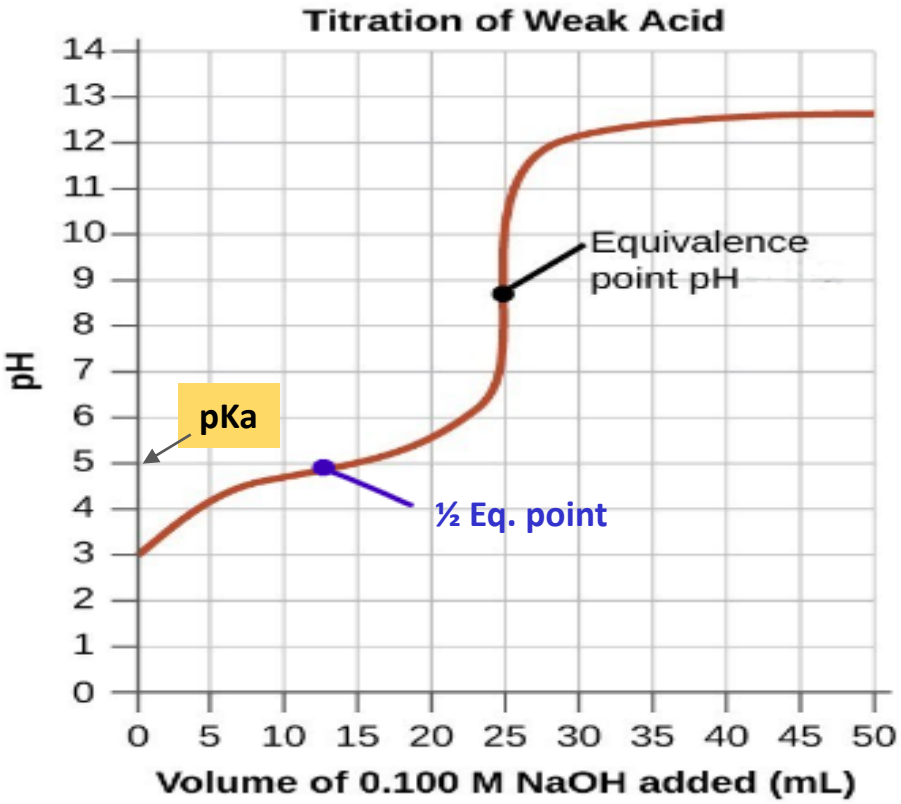
Titrate the solution with 0.1M NaOH solution in an increment of 1 or 2ml


Record the pH value after each addition in a draft paper.



*We will carry the titration twice to determine the equivalence point accurately*

Plot on a graph the number of mls of NaOH and the PH readings using Excel worksheet.



# GOOG LUCK