



B.PHARMA 1ST SEMESTER

PHARMACEUTICAL ANALYSIS-I

(BP102T)



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SECTION A

VERY SHORT ANSWERS TYPE QUESTIONS (10 × 2 = 20)

1. Describe the term normality.

Answer

Normality

- The normality of a solution is defined as the no. of gram equivalent present per litre of the solution. It is denoted by "N".

$$\text{Normality} = \text{No. of gram equivalents/Volume (in liters)}$$

2. Differentiate between Accuracy and Precision.

Answer

Accuracy	Precision
It is the closeness with the true value of the quantity being measured.	It is a measure of the reproducibility of the measurement.
Measurement can be accurate but not necessarily precise.	Measurement can be precise but not necessarily accurate.
It can be determined with a single measurement.	It needs several measurements to be determined.
Accuracy may be affected with systematic error.	Precision may be affected with random error.

3. Discuss the role of indicators in titrations.

Answer

Role of Indicators in Titrations

- Indicator is the substance that changes or imparts certain color at the stoichiometric end point of the titration. This means when a certain concentration of the acid or a base is completely neutralized, then at the end point we observe a color change.
- Hence, indicators in a titration are used to identify the equivalence or the end point of a reaction where pH change occurs.

4. Explain acid and base as per Arrhenius theory.

Answer

Acid and Base as Per Arrhenius Theory

- **Arrhenius acid**
 - In the aqueous solution increases the concentration of protons or H⁺ ions.
 - For example, hydrochloric acid in the water. HCl undergoes a dissociation reaction to produce an H⁺ ion and a Cl⁻ ion, as explained below. The concentration of the H⁺ ions is increased by forming hydronium ions.
- **Arrhenius base**
 - It is a substrate that increases the concentration of hydroxide ions in the aqueous solution.
 - The example for Arrhenius base is a highly soluble sodium hydroxide compound in water, which dissociates to give sodium ion and hydroxide ion.

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5. Define limit test.

Answer

Limit Test

- A limit test is a type of analytical test used to determine impurity in a substance whether a substance is present in a sample at a concentration above or below a specified limit. Limit tests are often used to ensure that a substance meets certain quality standards or regulatory requirements.

6. Define principle of gravimetry analysis.

Answer

Principle of Gravimetric Analysis

- The principle of gravimetric analysis is based on the estimation of the mass percent of an ion in an impure compound of known quantity by determining the mass of the same ion in a pure compound. In order to determine the mass, the ion of interest needs to be completely isolated.

7. Explore the term Dichrometry.

Answer

Dichrometry

- Primary standard used: Ammonium iron(II) sulphate
- Indicator used: N-phenyl anthranilic acid
- **PRINCIPLE:**
 - As an oxidant, dichromate has advantages over permanganate but it is less powerful, its use is much more limited.
 - It is obtainable in a state of high purity and can be used as primary standard solutions of dichromate in water are stable indefinite.
 - The half reaction for dichromate is:
$$\text{CrO}_7^{2-} + 14\text{H}^+ + 6\text{e}^- \rightarrow 2\text{Cr}^{3+} + 7\text{H}_2\text{O}$$
 - The most important application of dichromate is in its reaction with iron (II) in which it is often preferred to permanganate.
$$\text{Cr}_2\text{O}_7^{2-} + 6\text{Fe}^{2+} + 14\text{H}^+ \rightarrow 2\text{Cr}^{3+} + 6\text{Fe}^{3+} + 7\text{H}_2\text{O}$$

8. Write principle of Polarography.

Answer

Principle of Polarography

- This method of recording current flows between two electrodes, one of which is polarisable (the mercury-dropping electrode) and the other is nonpolarisable, by gradually increasing the voltage between them.
- The half-wave potential and diffusion current are calculated from a sigmoid shape current-voltage curve. Concentrations of substances are measured using diffusion currents. Every element possesses a half-wave potential.

9. What do you mean by electrochemical methods of analysis?

Answer

Electrochemical Method of Analysis

- Electroanalytical techniques are concerned with the interplay between electricity

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& chemistry, namely the measurement of electrical quantities such as current, potential or charge and their relationship to chemical parameters such as concentration.

- **Examples** – Polarography, Amperometry, Potentiometry.

10. Define metal ion indicator with suitable example.

Answer

Metal Ion Indicator

- A metal ion indicator is a substance that changes color when it binds to metal ions in solution.
- **Examples** – Murexide, Eriochrome black T.

SECTION B

LONG ANSWERS TYPE QUESTIONS (2 × 10 = 20)

1. Describe various types of errors and methods for minimizing them.

Answer

Types of Errors

1. Systematic, determinate or constant errors
2. Random or indeterminate errors
3. Errors in measurements
4. Gross errors
5. Other errors

1. Systematic, Determinate or Constant errors

- These errors can be avoided and their magnitude can be determined, thereby correcting the measurements.
- **Types of determinate errors:**
 - Personal errors
 - Operational errors
 - Instruments and reagent errors
 - Methodic errors
 - Additive and proportional errors

Errors	Description
Personal Errors	<ul style="list-style-type: none">➤ These errors are not connected with the method or procedure but the individual analyst is responsible for them.➤ This type of errors may arise due to the inability of the individual making observations.➤ Some important personal errors are:<ul style="list-style-type: none">○ Inability in judging colour change sharply in visual titrations.○ Error in reading a burette.

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	<ul style="list-style-type: none">○ Mechanical loss of material in various steps of an analysis.
Operational Errors	<ul style="list-style-type: none">➤ These errors are mostly physical occur when sound analytical technique is not followed.
Instruments and reagents Errors	<ul style="list-style-type: none">➤ Responsible for such errors: Following factors are:<ul style="list-style-type: none">○ Balance arms of unequal lengths.○ Uncalibrated or improperly calibrated weights.○ Incorrectly graduated burettes.
Methodic Errors	<ul style="list-style-type: none">➤ These are the most serious types of errors encountered in chemical analysis.➤ Some examples involving methodic errors are:<ul style="list-style-type: none">○ Solubility of precipitate in medium and in wash liquid.○ Decomposition or volatilization of weighing forms of precipitates on ignition or on heating.
Additive and Proportional Errors	<ul style="list-style-type: none">➤ Absolute error is independent of the amount of the constituent present in the determination e.g., loss in weight of a crucible.➤ On the other hand, the magnitude of proportional error depends upon the quantity of the constituent. e.g., impurity present in a standard substance.

2. Random or Indeterminate errors

- These errors are accidental and analyst has no control over them.
- They may be of two types :
 - 1. Variation within determinate errors** - These cannot be prevented from variation e.g., in igniting a precipitate of $\text{Al}(\text{OH})_3$ to constant weight, an analyst may obtain successive values, varying without a definite trend.
 - 2. Erratic errors** - Analyst has no control over such errors. Important examples of erratic errors are:
 - A. Vibration in balance while handling it.
 - B. Accidental loss of material during analysis.

3. Errors in Measurements

- Errors in weighing may be due to the insensitivity of balance.
- Wrong suspension of ring-riders.
- Placing the weights at edge of the pan.
- Using non-calibrated weights.
- Difference in temperature between the object weighed and the balance.

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4. Gross Errors

- Common gross errors are:
 - Use of numerically incorrect conversion factors.
 - Wrong selection of method.
 - Unsuitable storage of samples.

5. Other Errors

Besides above, there may be present some other types of errors, as:

1. errors in radiometric analysis
2. errors in chromatography
3. photometric errors.

Methods for Minimizing errors

1. Running a blank determination:

- Errors arising from the introduction of impurities through the reagents and vessels are accomplished by running a blank.
- Such a procedure involves going through all the analysis using the some solvent and reagent in the same quantities, but omitting the unknown component.
- Thus, in making a blank, sample is omitted, otherwise the details of the procedure are followed exactly as far as possible.

2. Calibration of apparatus and application of corrections:

- All instruments, such as burettes, pipettes, weights, measuring flasks, etc. must be properly calibrated and the appropriate corrections must be applied to the original measurements.

3. Running a controlled determination:

- It consists in carrying out a determination under identical experimental conditions as far as possible upon a quantity of a standard substance which contains the same weight of the constituent as is contained in the unknown sample.
- The weight of the constituent x in the unknown can then be calculated.

$$\frac{\text{Result found for standard}}{\text{Result found for unknown}} = \frac{\text{Wt. of constituent in standard}}{x}$$

4. Running of parallel determination:

- Parallel determinations serve as a check on the result of a single determination and indicate only the precision of the analysis.
- The values obtained in parallel determinations should agree well among themselves.
- These values should not vary by more than three parts per thousand.
- If larger variations are shown the determination must be repeated until satisfactory concordance is obtained.

5. Standard addition:

- A known amount of the constituent being determined is added to the sample, which is then analyzed for the total amount of constituent present.

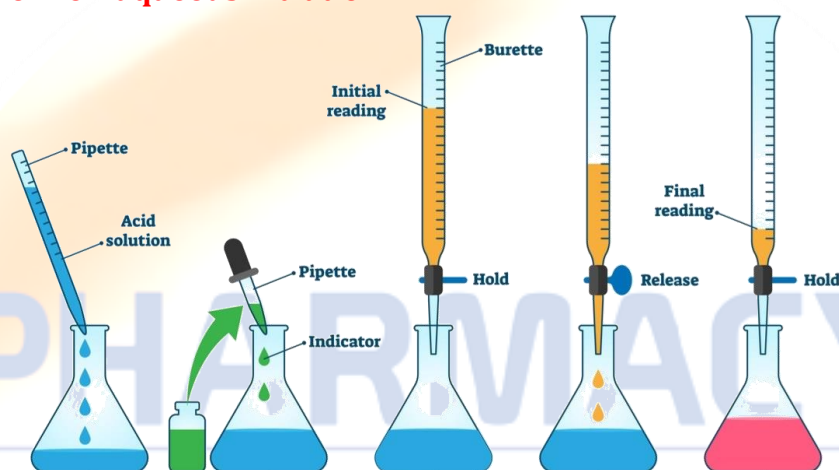
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- The difference between the analytical results for samples with and without the added constituent gives the recovery of the amount of added constituent.
- If the recovery is satisfactory, accuracy of the procedure is enhanced.
- This procedure is especially applied to physicochemical processes, as polarography and spectrophotometry.

2. Explain the significance of non-aqueous titrations. Differentiate between “Levelling solvents” and “Differentiating solvents” with suitable example.

Answer

Significance of Non-aqueous Titration



- Non-aqueous titration is used to titrate organic acids or bases that are insoluble in water and soluble in non-aqueous solvents.
- It can titrate weak acids or bases that are impossible to titrate in water.
- Organic acids with a strength comparable to water can be easily titrated.
- It can also be used to titrate samples that contain acid combinations.
- This method of titration is easy to carry out and does not require any specific laboratory equipment or glassware.
- Internal indicators can deliver a quick and precise result.
- This titration is crucial in pharmacopoeial assays and for biological substances.
- The potentiometric approach can be used to automate the operation.

Difference between Levelling solvents and Differentiating solvents

Levelling Solvents

- Levelling solvent or levelling effect is the effect of a solvent on the properties of acids and bases.
- For example, the strength of a strong acid can be limited or levelled by the basicity of the solvent. Therefore, we can level the strength of a strong base by the acidity of the solvent.
- Typically, a strong acid dissolves in water and forms hydronium ions upon reaction with water molecules.
- Acids that are stronger than hydronium ions and tend to react with water molecules to form hydronium ions. In other words, strong acids that are stronger than hydronium ions cannot exist in water.

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- E.g. aqueous perchloric acid can completely ionize in water, and it is a strong acid.
- Generally, strong bases are considered as leveling solvents for acids. When there is a leveling solvent, many acids tend to completely dissociate into ions, and therefore, they have the same strength.
- Moreover, all acids become indistinguishable in strength when they are dissolved in strongly basic solvents due to their greater affinity of the strong bases for protons. We call this phenomenon the leveling effect.

Differentiating Solvents

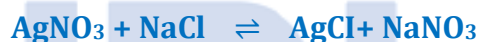
- Differentiating solvents are chemical solvents that cause various acids to dissociate to different degrees.
- In other words, acids dissociate partially in differentiating solvents. Therefore, acids have different strengths when they are in these solvents.
- E.g., anhydrous acetic acid is a solvent and a weak proton acceptor than water.
- Therefore, strong acids such as hydrochloric acid partially dissociate into ions when they are in this type of solvents. Strong acids show different strengths when they are in this type of solvent.

3. Discuss the detailed account of Mohr's method and Volhard's method.

Answer

Mohr's Method

- "Mohr's method is a titration between silver nitrate and halides in which potassium chromate is used as indicator".



- Principle:
- When AgNO_3 is added in Cl solution (example NaCl) containing K_2CrO_4 , then two reactions takes place:



- K_{sp} of AgCl and Ag_2CrO_4 are 1×10^{-10} and 9×10^{-12} respectively.
- A substance having smaller K_{sp} value is precipitated before substance having larger K_{sp} value.
- Thus AgCl ($K_{sp} = 1 \times 10^{-10}$) is precipitated before Ag_2CrO_4 ($K_{sp} = 9 \times 10^{-12}$) and white precipitate is observed in titration vessel.
- As more AgNO_3 is added, concentration of Cl decreases and when $[\text{Cl}^-]$ becomes equal or less than $(\leq) \sqrt{\frac{CrO_4}{2.5 \times 10^6}}$
- Then, reddish brown precipitate (Ag_2CrO_4) is formed to indicate the end point.

Experimental details:

Step 1. Preparation of AgNO_3 solution

AgNO_3 is a solid secondary standard. Using formula: weight of AgNO_3 is calculated, weighed, dissolved in distilled water & volume is made up.

$$\frac{\text{molarity} \times \text{molecular weight} \times \text{volume in ml}}{1000}$$

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Step 2. Standardization of AgNO₃ solution

a. Preparation of NKCl solution: NKCl is a solid primary standard. Using formula; weight of Cl is calculated, weighed, dissolved in distilled water & volume is made up.

$$\frac{\text{molarity} \times \text{molecular weight} \times \text{volume in ml}}{1000}$$

b. Preparation of potassium chromate solution (2%): 2 g of K₂CrO₄ is dissolved in water and volume is made upto 100 ml.

c. Titration: 10 mL of KCl solution & 1 ml of K₂CrO₄ solution are taken in a conical flask. To that solution AgNO₃ solution is added until reddish brown precipitate (Ag₂CrO₄) is formed.

Step 3. Calculation of molarity determination

$$M_{\text{AgNO}_3} V_{\text{AgNO}_3} = M_{\text{KCl}} V_{\text{KCl}}$$
$$M_{\text{AgNO}_3} = \frac{M_{\text{KCl}} V_{\text{KCl}}}{V_{\text{AgNO}_3}}$$

Limitations / Precautions of Mohr's Method

- Mohr's method is used only in neutral medium.
 - Ag₂CrO₄ is soluble in acid.
 - Base precipitates the AgNO₃ as Ag(OH)₂. Thus acidic or basic medium is neutralized before titration.
- Mohr's method is not used for iodides. AgI and Ag₂CrO₄ both have reddish brown colour and thus end point cannot be detected.

Volhard's Method

Experimental details:

- **Step 1. Preparation of NaSCN solution:** NaSCN is a solid secondary standard. Using formula; weight of NAsCN is calculated, weighed, dissolved in distilled water & volume is made up.

$$\frac{\text{molarity} \times \text{molecular weight} \times \text{volume in ml}}{1000}$$

- **Step 2. Titration with AgNO₃ solution**

a. Preparation of AgNO₃ solution: Using formula; weight of AgNO₃ is calculated, weighed, dissolved in distilled water & volume is made up.

$$\frac{\text{molarity} \times \text{molecular weight} \times \text{volume in ml}}{1000}$$

b. Preparation of ferric alum solution (5%) : Dissolve 5 g of ferric ammonium sulphate in a mixture of 30 ml of water and 0.6 ml of sulphuric acid. Dilute with sufficient freshly boiled and cooled water to produce 100 ml.

c. Titration: 10 mL of AgNO₃ solution, 6N HNO₃ & 1 ml of ferric alum solution are taken in a conical flask. To that solution NaSCN solution is added until reddish brown coloured solution (due to Fe(SCN)₃) is formed.

- **Step 3. Calculation**

$$M_{\text{NaSCN}} V_{\text{NaSCN}} = M_{\text{AgNO}_3} V_{\text{AgNO}_3}$$

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$$M_{NaSCN} = \frac{M_{AgNO_3} V_{AgNO_3}}{V_{NaSCN}}$$

Limitations/Precautions of Volhard's Method

1. Volhard's method is used only in acidic medium. Nitric acid help in coagulation of precipitation and prevent hydrolysis of ferric alum indicator.
2. Volhard's method cannot be used for analysis of halides directly.

SECTION C

SHORT ANSWERS TYPE QUESTIONS (5 × 7 = 20)

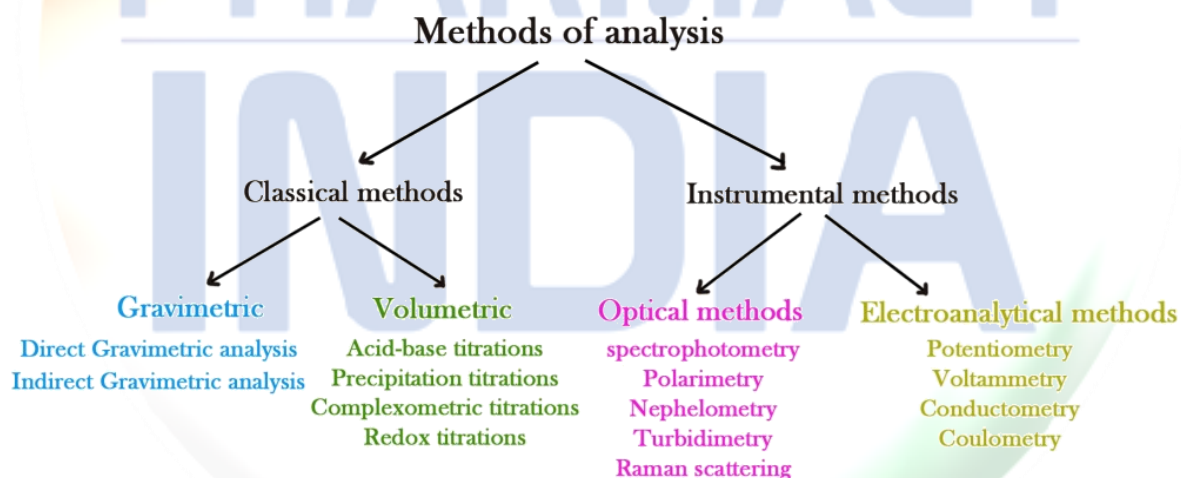
1. Outline the various techniques of analysis used in pharmaceuticals.

Answer

Various Techniques of Analysis used in Pharmaceuticals

1. Chemical Methods
2. Physico-chemical Methods
3. Microbiological Methods
4. Biological Methods

1. Chemical Methods



1. Titrimetric Analysis (also termed as volumetric analysis)

- In this technique the substance to be determined is allowed to react with an appropriate reagent added as a standard solution, and the volume of solution needed for completion on reaction is determined.
- Following are the types of titrimetric analysis:
 - (i) Neutralization (acid-base) reactions
 - (ii) Complexometric titrations
 - (iii) Precipitation titrations
 - (iv) Oxidation-reduction titrations
 - (v) Non-aqueous titration

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- Neutralization/aqueous Acid-Base Titrations:** It involves neutralization reaction in presence of water as solvent.
- Non-aqueous Titrations:** It involves the reaction between acid & base in presence of non-aqueous i.e. organic solvents.
- Precipitation Reactions:** It involves the reaction leading to precipitate formation. It includes the methods where the reacting substance & standard solution react to yield a precipitate or a slightly soluble salt as the primary reaction product.
- Complexometric Titrations:-** It includes all the methods wherein the reacting substance & the standard solution react to form a soluble but very slightly dissociated complex substance. It is based on complex formation reaction mainly EDTA titrations.
- Redox Titrations:-** These titrations involve simultaneous oxidation reductions reactions. It includes all the methods wherein reacting substance is oxidized or reduced by the standard solution.

2. Gravimetric Analysis

- In this technique substance under determination is converted into an insoluble precipitate which is collected and weighed.
- In a special case of gravimetric analysis, electrolysis of the substance is carried out and the material deposited on one of the electrodes is weighed, this technique is called as electrogravimetry.

2. Physicochemical Methods

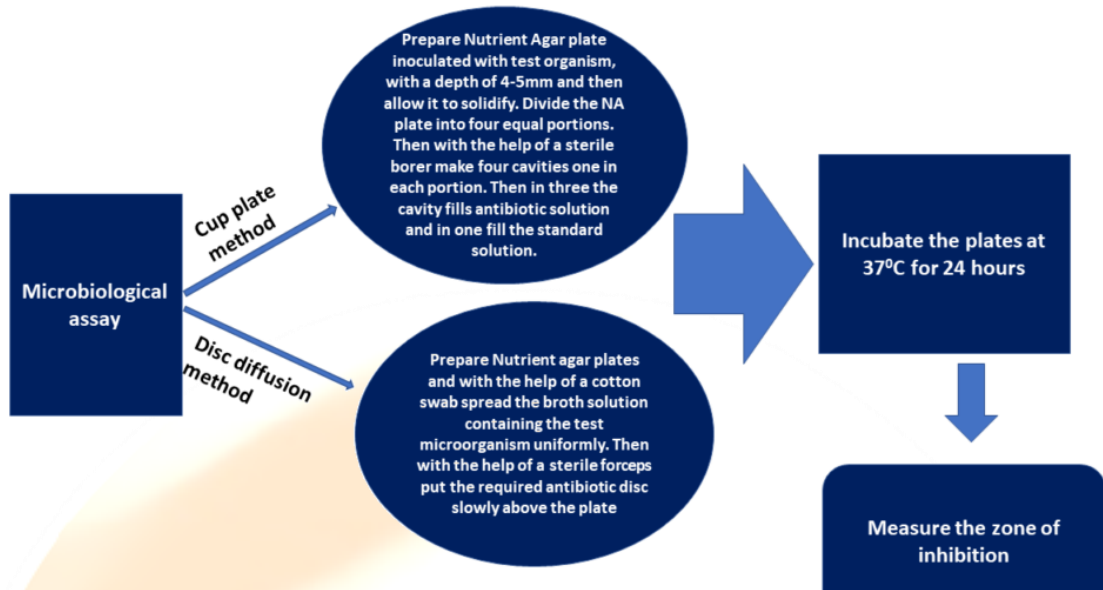
Physical Properties	Instrumental Methods
Electrical potential	Potentiometer
Electrical conductance	Conductometry
Electrical current	Paleography, Voltametry
Absorption of radiation	Spectrophotometry, Colorimetry, Atomic absorption spectroscopy
Emission of radiation	Emission spectroscopy, Flame photometry, Fluorimetry
Scattering of radiation	Turbidimetric, Nephelometry
Refraction of radiation	Refractometry
Rotation of plane of Polarized light	Polarimetry, Optical rotatory dispersion
Thermal properties	Thermal method
Mass to charge ratio	Mass spectrometry

3. Microbiological Methods

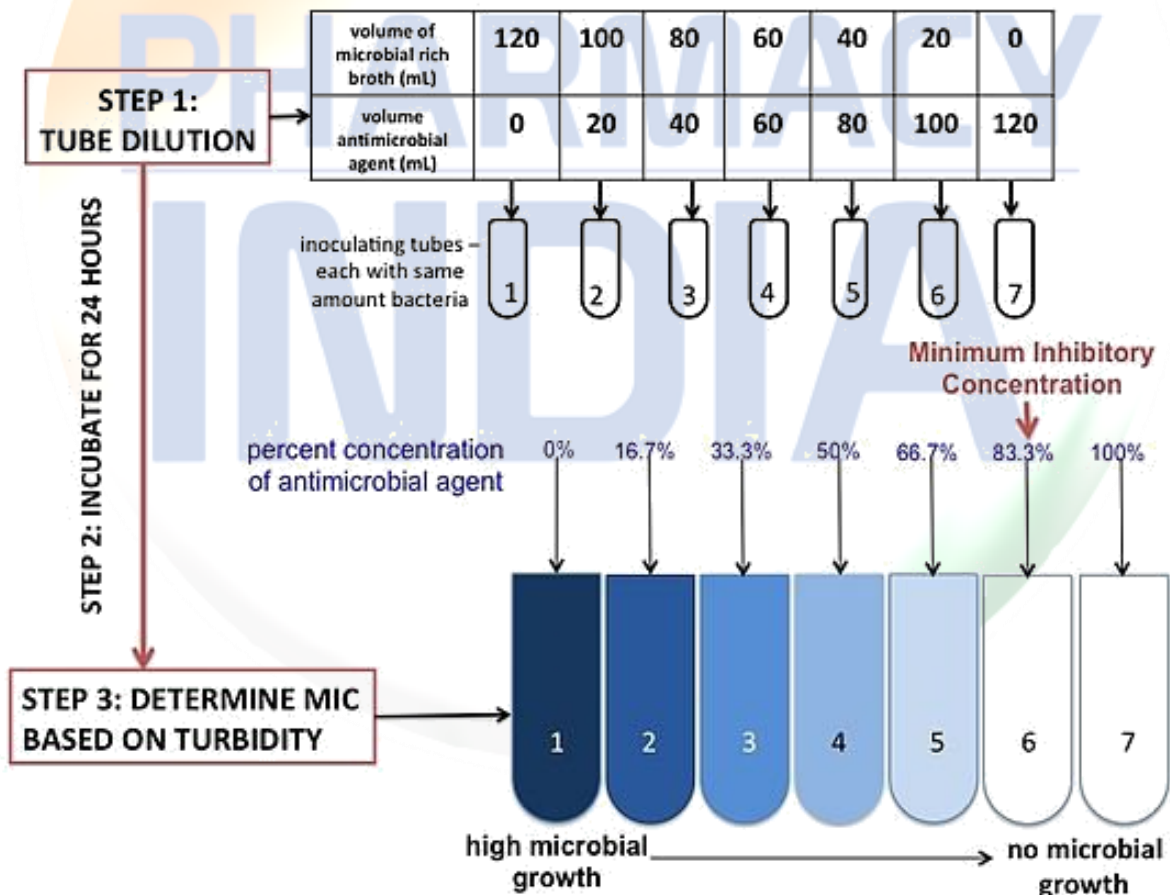
1. Cylindrical Plate or Cup plate method
2. Turbidimetric or Tube assay method

Cylindrical or Cup Plate method

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Turbidimetric or Tube assay method



2. Discuss various neutralization curve of acid base titrations.

Answer

Neutralization Curve of Acid Base Titrations

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- A plot of pH against the volume of alkali added (mL) is known as a neutralization or titration curve.
- The ideal pH range for an indicator is 4.5-9.5.
- Types
 - Strong acid (HCl) vs strong base (NaOH)
 - Weak acid (CH₃COOH) vs strong alkali (NaOH)
 - Strong acid (HCl) vs weak base (NH₃)
 - Weak acid (CH₃COOH) vs weak base (NH₃)

A. Strong acid (HCl) vs strong base (NaOH)

- When a strong acid and strong base are mixed together then reaction occurs are represented as follows



- Since, it is a Reverse process of ionization of water
- The calculation of titration curve is illustrated as consider the titration of 100ml, N HCL with N NaOH. The pH before titration commences is pH of N HCL. Therefore,

$$(\text{H}^+) = 1\text{N or PH} = 0$$

- After adding 1 ml of titrant the volume becomes 101 ml and it contains (100 - 1) = 99 ml equivalent of acid in 101 ml or 99/101 eq/liter.

$$[\text{H}^+] = 99/101$$

So,

$$\text{PH} = -\log (99/101)$$

$$\text{PH} = \log (101/99) = \log 1.020$$

$$\text{PH} = 0.0086$$

- Then after addition of 90ml titrant

$$(\text{H}^+) = 10/190$$

$$\text{PH} = 2.2788$$

- After addition of 99.9 ml of titrant

$$(\text{H}^+) = 0.1/199.9$$

$$\text{PH} = 3.3008$$

- Then after the addition of a 100ml titrant, the pH will change sharply to the equivalence point.

- Then after the addition of a 100ml of titrant

$$[\text{OH}] = 1/201$$

$$\text{P}[\text{OH}] = \log(201/1)$$

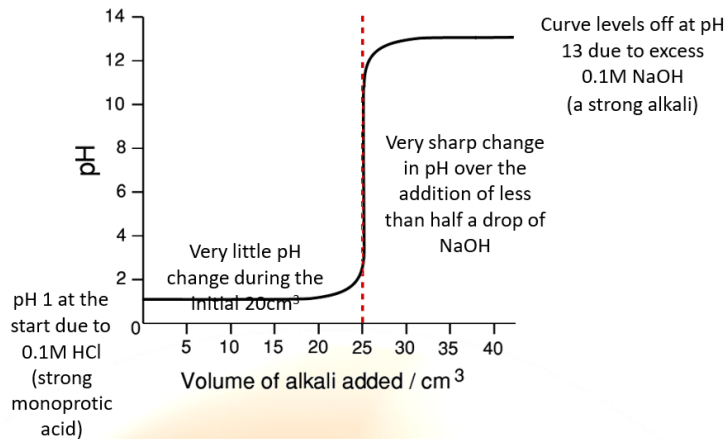
$$\text{p}[\text{OH}] = 2.3032$$

$$\text{PH} = 14 - \text{pOH}$$

$$\text{PH} = 11.6968$$

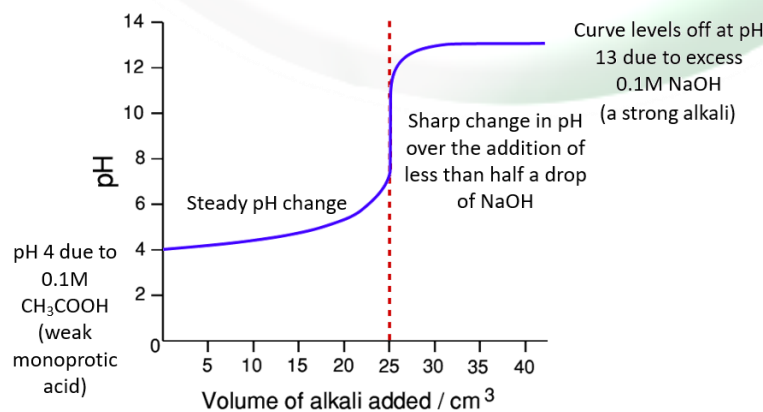
- As the titration process, the pH Rises slowly, but near the equivalence point the change in pH is very sharp 3.3 (or 2.2988) to 7 so, with 1N solution in indicator which is effective in pH range 3 to 10.5 may be used. e.g phenolphthalein, methyl orange, methyl red, phenol red.

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B. Weak acid (CH₃COOH) vs strong alkali (NaOH)

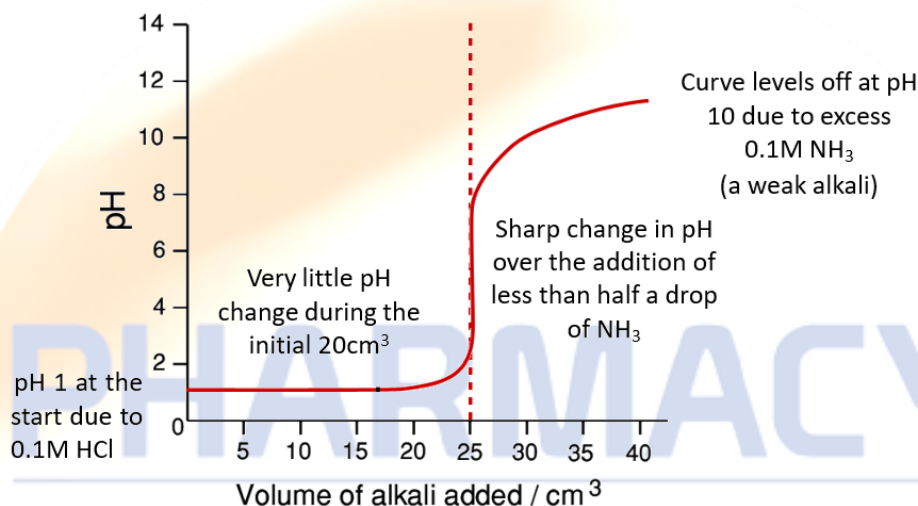
- Titration is represented by the following equations
 $HA + OH \rightleftharpoons H_2O + A$
 where HA is A weak acid and it is in undissociated form,
 - This titration is different from the first type of titration i.e., strong acid strong base titration.
- By three ways that ways of differences described as follows
- The H concentration is small at the start of the titration because of un dissociation but pH will be greater.
 - In 0.1 NHCL (H^+) = 0.1 and PH = 3.127
 - In 0.1 N CH₃ COOH is a weak acid and is poorly dissociated, hence it gives a smaller concentration of H⁺ ions.
 - A solution is added to the weak acid the salt form produces a common Ion effect so that the dissociation of a weak acid is suppressed. e.g., when NaOH is added to CH₃COOH there is a formation of salt and the following equilibria exist.
 $CH_3COOH \rightarrow H^+ + CH_3COO^-$ (poorly dissociation)
 $CH_3COONa \rightarrow Na^+ + CH_3COO^-$ (strongly dissociation)
 - In the titration of HCl with NaOH, PH at the equivalence point is 7 but if a weak acid like CH₃COOH is taken pH at the completion of titration is more than 7.
 $CH_3COONa + H_2O \rightarrow CH_3COOH + Na^+ + OH^-$
 - Due to the hydrolysis of West minus is greater than h + in the solution is alkaline basic e.g. its pH is greater than 7.



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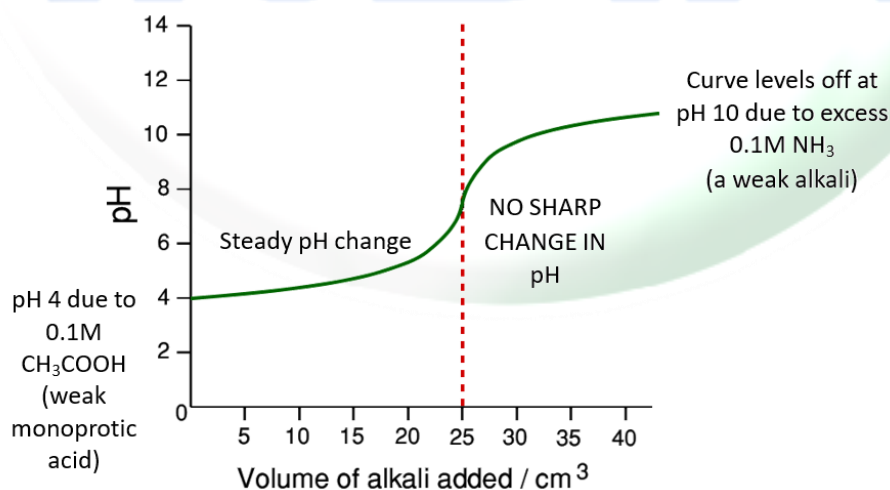
C. Strong acid (HCl) vs weak base (NH₃)

- Here we consider the titration of 0.1 M and 0.1 N aq. Ammonia solution with 0.1 M and 0.1 N HCl solution.
- The reaction involved
 $\text{NH}_4\text{OH} + \text{HCl} \rightarrow \text{NH}_4\text{Cl} + \text{H}_2\text{O}$
- The salt NH₄Cl is hydrolysis and $\text{NH}_4\text{OH} + \text{H}_2\text{O} \rightarrow \text{NH}_4\text{OH} + \text{H}^+$
- Due to the formation of H⁺ ion, the pH at the equivalence point will be slightly on the acid side as shown by the following calculate.
- Suppose we titrate 50 ml of 0.1 M NH₄OH with 0.1M HCl.



D. Weak acid (CH₃COOH) vs weak base (NH₃)

- Let Us consider the titration of 0.1 M CH₃COOH with 0.1N NH₄OH
 $\text{CH}_3\text{COOH} + \text{NH}_4\text{OH} \rightarrow \text{CH}_3\text{COONH}_4 + \text{H}_2\text{O}$
- The salt formed, CH₃COONH₄ undergoes hydrolysis
 $\text{CH}_3\text{COONH}_4 + \text{H}_2\text{O} \rightarrow \text{CH}_3\text{COOH} + \text{NH}_4\text{OH}$



3. Differentiate co-precipitation and post precipitation with suitable example.

Answer

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- The rate of coprecipitation is always greater than the rate of post precipitation.
- In coprecipitation, two or more ions get together and form an insoluble compound whereas in post precipitation only one ion is involved.
- Coprecipitation is a reversible process but post precipitation is not.
- The driving force behind coprecipitation is hydrophobic interactions whereas in post precipitation it is electrostatic interactions.
- In coprecipitation, the precipitate is formed in the solution phase itself whereas, in post precipitation, the precipitate is formed in the gas phase.
- The morphology of the precipitate formed by coprecipitation is more complex as compared to the precipitate formed by post precipitation.
- The difference between post precipitation and coprecipitation is that the former involves a solid-solid interaction, while the latter does not. In coprecipitation, the two substances interact to form a precipitate. In post precipitation, the solid and liquid phases are in equilibrium with each other.
- The contamination degree is higher in coprecipitation than post precipitation
- The time required for the completion of the process is less in coprecipitation as compared to post precipitation.
- The rate of diffusion is more in coprecipitation than post precipitation.
- Coprecipitation involves a chemical reaction while post precipitation does not.
- The difference between the two mechanisms is that coprecipitation results in a more stable product. This is because the precipitate formed during coprecipitation is more likely to be insoluble. In contrast, post precipitation results in a less stable product. This is because the precipitate formed during post precipitation is more likely to be soluble.

4. Explain Iodometry and Iodimetry.

Answer

Iodometry

DEFINITION: The titrations in which the equivalent amount of I_2 is liberated from KI by the sample the liberated I_2 is titrated against standard $Na_2S_2O_3$ solution such a type of indirect determination of strong oxidizing agents is called iodometry.

PRINCIPLE: The titrations in which the liberated I_2 is titrated with a standard solution of $Na_2S_2O_3$ solution using starch-mucilage as indicator is called iodometric titrations. The indirect iodometric titration method is termed as iodometry.



STANDARDIZATION OF 0.1M $Na_2S_2O_3$:

1. **Preparation of 0.1M $Na_2S_2O_3$ solution:** Dissolve 25gm of $Na_2S_2O_3$ and 0.2gm of Na_2CO_3 in CO_2 free water and dilute to 1000ml with water.
2. **STANDARDIZATION OF $Na_2S_2O_3$ SOLUTION:** Dissolve 0.2gm of $KBrO_3$ weighed accurately in sufficient water to produce 250ml. To 50ml of this solution add 2gm of KI and 3ml of 2M HCl solution and titrate with $Na_2S_2O_3$ solution using starch solution as indicator until the blue color disappears.

IP FACTOR: Each ml of 0.1M $Na_2S_2O_3$ solution = 0.002784gm of $KBrO_3$.

CALCULATION: M of $Na_2S_2O_3$ solution = $\frac{\text{Wt. of } KBrO_3 \text{ taken} \times \text{Required Molarity}}{\text{Titration vol} \times \text{IP factor}}$

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Iodimetry

DEFINITION: Determination involving direct titration with iodine due to oxidizing powder of iodine in aqueous solution such type of titrations are called iodimetric titrations.

PRINCIPLE:

- Oxidation and reduction process involving I_2 is called in general as iodimetric titrations. The direct titration with a standard solution of I_2 is termed as iodimetric. Solutions of I_2 are weak oxidizing agents that are used for the determination of strong oxidizing agents. The most accurate description of the half reaction for I_2 in the application is :



Where I_3^- is the tri-iodide ion.

- Standard I_2 solutions have relatively limited applications compared with the other oxidants because of their significantly smaller electrode potential. A solution of I_2 in aqueous media has an intense yellow to brown color. Since I_2 solution can serve as its own indicator (self- indicator). However in practice starch solution is used as an indicator for the most accurate end point.

STANDARDIZATION OF 0.05M I_2 USING As_2O_3 :

1. **PREPARATION OF 0.05M I_2 SOLUTION:** Dissolve about 14gm of I_2 in a solution of 36gm of KI in 100ml of water, add 3 drops of HCl and dilute with water to 100ml.
2. **STANDARDIZATION OF I_2 SOLUTION:**
 - Weigh accurately about 0.15gm of As_2O_3 , previously dried at 105° for 1 hour and dissolve in 20ml of 1M NaOH by warming, if necessary.
 - Dilute with 40ml of water, add 0.1ml of methyl orange solution and add drop wise dilute HCl until the yellow color is changed to pink.
 - Add 2gm of $NaHCO_3$, dilute with 50ml of water and add 3ml of starch solution as indicator.
 - Titrate with prepared I_2 solution until permanent blue color is produced.

IP/ EQUIVALENT FACTOR: Each ml of 0.05M I_2 solution = 0.004946gm of As_2O_3 .

CALCULATION:

$$M \text{ of } I_2 \text{ solution} = \frac{\text{Wt. } As_2O_3 \times \text{Expected M}}{\text{Titration vol} \times \text{IP factor}}$$

5. Discuss estimation of Barium sulphate.

Answer

Estimation of Barium Sulphate

- 0.5g of Sodium sulphate was weighed accurately and dissolved in 100ml water in a volumetric flask.

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- About 5ml 2N HCl was added.
- The solution is heated to boiling and a hot solution of BaCl₂ was added drop by drop with constant stirring, till the precipitation is complete.
- The solution containing the precipitate is heated in a water bath for 5 minutes. The precipitate is allowed to stand for an hour.



- The clear solution is decanted through an ashless filter paper (whatman No. 40).
- The precipitate is washed with hot distilled water to free sulphate ions. The particles adhering to the sides of the beaker and glass rod are removed by a policeman.
- Finally the precipitate is washed once again. The dried filter paper is folded and placed in a crucible which has been previously weighed.
- The filter paper with the precipitate is first incinerated on a Bunsen burner by a low flame and then transferred to an electric burner.
- The crucible is then transferred to a desiccator and cooled. When cold, the crucible is weighed.

Calculation:

- Mass of crucible + lid = 23 g
- Mass of crucible + lid + Barium sulphate = 24 g
- Mass of Barium sulphate = (b-a) = 1 g.
- 233.36 of barium sulphate contain 137.36 g of barium.
- Mass of barium in 1 g of Barium sulphate = $(137.36 * 1)/233.36$
- Therefore, Mass of Barium in the whole of the given solution = 0.58g
- Result: Mass of Barium in the whole of the given solution = 0.58 g

6. Illustrate the principle, instrumentation and applications of conductometry.

Answer

Conductometry

Principle

- Based on the conductance of electrical current through electrolyte solutions similar to metallic conductors.
- The electric conductance in accordance with ohms law which states that the strength of current(i) passing through conductor is directly proportional to potential difference & inversely to resistance.

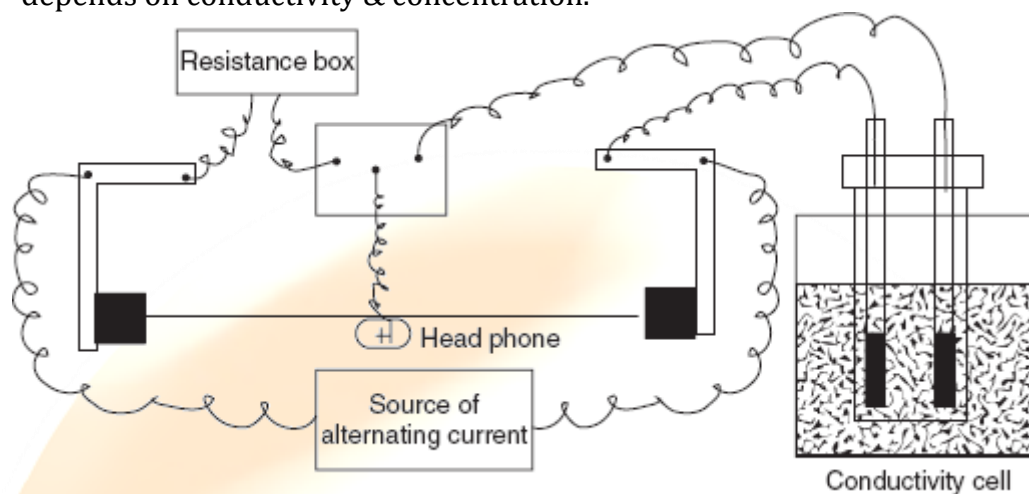
$$i = V/R$$

Instrumentation

- **Current source :-** Alternative current source is used. High frequency alternating current generator is employed. electrical potential is applied ions will be transfer and ultimately conductance take place.
- **Conductivity meter :-** Digital display, calibrator, power switch.
- **Conductivity cell :-** Made of pyrex or quartz and are fitted with two platinum electrodes. Should be placed in vessel containing water to maintain constant temp. types of conductivity cells are TYPE-A, TYPE-B, & TYPE-C.

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- **Electrodes :-** Platinum sheets each of 1 cm are fixed at distance of 1 cm. the surface is coated with platinum black to avoid polarization effect & increase effective surface area. platinisation of electrodes is done by coating solutions 3% chlorplatinic acid & lead acetate on it to gel uniform coating. electrode usage depends on conductivity & concentration.



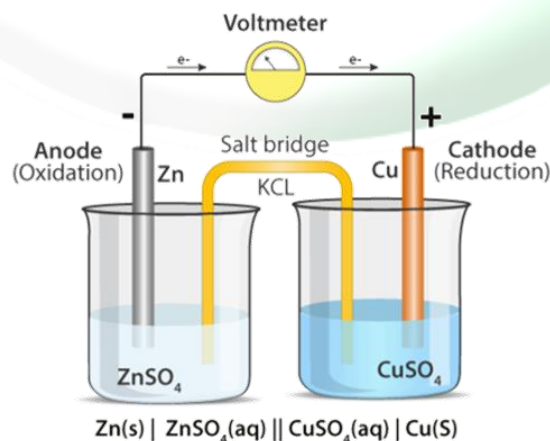
Applications

1. Solubility determination of sparingly soluble compound
2. **Kinetics study:** Reaction progress can be judged by measuring the conductance before, during and at the end of the reaction.
3. Degree of dissociation
4. Basicity of organic acid
5. **Determination of concentration**
 - Calibration curve is plotted between concentration of standard solutions and their conductance.
 - Conductance of sample is measured and its concentration is determined from the calibration curve.

7. Draw the construction of electrochemical cell. Describe the working of standard hydrogen electrode and standard calomel electrode.

Answer

Construction of Electrochemical Cell



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Electrochemical cells generally consist of a cathode and an anode.

Cathode

- Denoted by a positive sign since electrons are consumed here
- A reduction reaction occurs in the cathode of an electrochemical cell
- Electrons move into the cathode

Anode

- Denoted by a negative sign since electrons are liberated here
- An oxidation reaction occurs here
- Electrons move out of the anode

Working of Standard Hydrogen electrode

- Standard Hydrogen Electrode can work both as an anode and as a cathode.
- When a standard hydrogen electrode undergoes oxidation in a cell, it acts as an anode. During this process, hydrogen gets changed into H^+ ions which go into the solution.



- When a standard hydrogen electrode undergoes a reduction in a cell, it acts as a cathode. During this process, H^+ ions from the solution change into hydrogen gas.



- Thus, the electrode is reversible concerning H^+ ions.
- The electrode potential of the standard hydrogen electrode is zero at 298K.

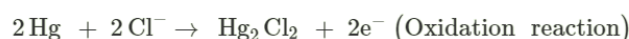
Working of Standard Calomel Electrode

- The calomel electrode is the mercury-mercurous chloride electrode.
- It consists of a glass vessel having a bent side tube.
- Pure mercury is placed at the bottom of the tube, which is covered with a paste of mercury-mercurous chloride ($Hg^+Hg_2Cl_2$) that is calomel.
- The remaining portion of the cell is filled with a solution of normal (1N) or saturated KCl (Potassium chloride).
- A platinum wire sealed into a glass tube is dipped into a mercury layer and is used to provide external electrical contact.
- The side tube is used for making electrical contact with a salt bridge.
- The calomel electrode can act as an anode or cathode depending on the nature of another electrode of the cell.

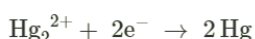
Reaction at the anode:



Overall reaction:



Reaction at the cathode:



Overall reaction:



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