Model Question and Answer

B. Pharm 1ST Year 1ST SEM

Subject- Pharmaceutical Analysis Subject Code-BP102T

UNIT - I

Long question (10 marks)

1. what is error? classify them and describe the method of minimizing error. Introduction of Errors— Errors is defined as the deformity present in any measurements by addition of any internal or external factor.

In the pharmaceutical science errors are induced by the defective equipment and methods.

In analytical chemistry errors are affects the material products reliability, reproducibility, and accuracy or precision.

During an analysis, the results are expected to be highly accurate and precise. However, it is not happening in all cases because due to presence of errors.

These errors may be predictable or unpredictable. Depending on the calculative nature errors are categorized into two parts.

Absolute Errors— Difference between experimental mean value and true/actual value is known as absolute errors. Absolute errors may be positive and negative.

Absolute errors = Measures mean value- True value

Relative Errors— Relative error is defined by dividing the absolute errors by the true values.

It is generally expressed as percentage, that is by the multiplying the relative error by 100 or by expressing it as parts per thousand by multiplying the relative error by 1000.

TYPES OF ERROR

1. Determinate (systematic) errors

2. Indeterminate (random) errors

1. Determinate errors-

Determinate or Systematic Errors—

Systematic errors are arising due to the wrong procedure, wrong measurement (pipettes, burette, volumetric flasks) and faulty instruments (calibrated balance and machinery system). Systemic error is under the control of the analyst because it is easily detectable and can be eliminated to a large extent.

Sources of systematic Errors—

sources are mentions below.

Instrumental Errors— Due to the use of defective equipment or low-quality instruments, errors are arising in the analytical procedure. It is easily checkable by the analyst.

Proportional Errors— The absolute value of this kind of the errors changes with the size of the sample in such a fashion that the relative error remains constant. It is easily incorporated by a material that directly interferes in analytical process.

Personal Errors— Errors are induced due to the carelessness, or ignorance and lack of skilled. This error is also called operative error. It is occurring by who are handling the method of analysis.

Chemical/Reagent Errors— Chemical errors are based on the chemical reactivity between the using chemical and reagent.

Errors in Methodology— It is a most serious error in analysis, as the error arises due to faulty methods.

2. Indeterminate errors-

Indeterminate or Random Errors— In this error we not define the specific well-known reason and cannot be eliminated, so it is also called as accidental errors.

• It is induced by the several successive measurements performed by the same analyst under the same conditions and identical experiments.

Such accidental errors will follow a random distribution pattern and the mathematical law of probability can be applied to get net conclusion regarding the results.

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How to minimizing the errors—

- Calibration of Instruments, apparatus.
- Personal care (skilled) required.

- Choosing the suitable and usable materials.
- Exhausted the impurities contamination.
- Study chemical evaluation and analysis.
- Proper methodology.

2. **Discuss the preparation and standardization of the potassium permanganate solution. . Preparation of 0.1 N Potassium Permanganate Solution:**

- **Weighing:** Accurately weigh the required amount of potassium permanganate crystals ($KMnO_4$) using an analytical balance. The molar mass of potassium permanganate is 158.034 g/mol.
- **Dissolution:** Dissolve the weighed potassium permanganate crystals in distilled water in a suitable container. Stir the solution thoroughly to ensure complete dissolution. Potassium permanganate dissolves in water to give a purple solution.
- **Volumetric Adjustment:** Transfer the solution to a volumetric flask of known volume, such as a 1000 mL flask. Rinse the weighing container with distilled water to ensure all the potassium permanganate is transferred to the flask. Then, fill the flask to the mark with distilled water, using a dropper or a funnel for precision.
- **Mixing:** Cap the flask and invert it several times to ensure homogeneity. This results in a 0.1 N potassium permanganate solution, where the

normality is precisely defined by the molarity and the volume of the volumetric flask.

2. Standardization of Potassium Permanganate:

To standardize the potassium permanganate solution, perform a titration with a primary standard reducing agent, such as oxalic acid ($H_2C_2O_4$).

- **Weighing Oxalic Acid:** Accurately weigh a known amount of oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$) using an analytical balance. The molar mass of oxalic acid dihydrate is 126.07 g/mol.
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- the oxalic acid solution. Record the volume of potassium permanganate required to react with the oxalic acid completely.
- **Calculation:** Use the stoichiometry of the reaction between potassium permanganate and oxalic acid to calculate the exact normality of the potassium permanganate solution. The balanced chemical equation for the reaction is:
- $5H_2C_2O_4 + 2KMnO_4 + 3H_2SO_4 \rightarrow 2MnSO_4 + 10CO_2 + 8H_2O + K_2SO_4$.
- **Repeat:** Repeat the titration if necessary to ensure accuracy and precision.

3. Importance:

The preparation and standardization of the 0.1 N potassium permanganate solution are essential in analytical chemistry. The accurate determination of normality ensures reliable and consistent results when this solution is used in various titrations or analytical procedures, particularly in the quantitative analysis of reducing agents.

In summary, the meticulous preparation and standardization of the 0.1 N potassium permanganate solution involve precise measurements, dissolution,

volumetric adjustments, and subsequent titration with a primary standard, ensuring the accuracy of the solution's normality for analytical applications.

3. Write down the source and effect of impurities.

IMPURITIES IN PHARMACEUTICAL MEDICINAL AGENT

Impurities in pharmaceuticals Impurities —

Impurities is defined as the presence of undesired/unexpected material during any procedure and may alters the final products.

The substances that are used in the pharmaceuticals should be pure enough to be used safely but it is difficult to obtain an absolute pure substance.

Generally, the impurities is the accidental factors and some time it is depends on the several method of the manufacture, and types of crystallization or purification process.

Most of impurities cause the harmful effect in the pharmaceutical preparation so it is a challenging task for pharmaceutical to removing the impurities.

Alternatively, a reasonably acceptable purity can be achieved by controlling various sources or reasons that add to the impure nature of an active pharmaceutical ingredients, or drugs as well as excipients used in pharmaceutical formulations. pharmacopoeia have fixed the limit for their impurities.

is likely to contain Barium as an impurity.

Solvents used in the process of purification— Often the solvents used for purification can be sources of impurities. These solvents range from organic solvents to acid (organic as well as mineral) and of course water.

Sources of impurities. Impurities may enter or formed in a drug substance during any of the following three stages—

- 1. During manufacturing.

2. During purification and processing.

3. During storage.

1. During manufacturing Raw material employed—`

Impurities present in raw materials may be carried through the manufacturing process to contaminate the final product. Impurities such as , Pb, Heavy metals, chlorides associate in the manufacturing unit.

Example- Rock salts contain the small amount of calcium sulphate and magnesium chloride. Thus, sodium chloride prepared from this source will contain traces of calcium and magnesium compounds.

Example- Copper sulphate may be prepared by the action of sulphuric acid on copper turnings. Copper turning are known to have iron and arsenic impurities.

Reagents used in manufacturing process-

The quality and purity of reagents used for manufacturing the drug substances are very important. If reagent used in the manufacturing process contains some impurities these may find entry into the final products

Example- Sulphuric acid is used in many chemical processes.

This acid often has lead present in it. Anions like chlorine and sulphate are common impurities in many substances because of the use of hydrochloric acid and sulphuric acid respectively in processing.

Solvents used in the manufacturing process-

Naturally, solvents play an important role next to the main reagents as most of the chemical reactions involved in these processes are solvent based. If proper quality/purity of solvents is not assured, they may add to the impurities. Solvents like toluene, n-butanol contain water as an azeotrope. Alcoholic solvents also may be contaminated with water and ethyl acetate can contain acetic acid in small amounts. Thus, quality of solvents needs to be assured and controlled.

Reaction equipment-

The reaction vessels employed in the manufacturing process may be metallic or mild steel with glass lining. Some solvents and reagents employed in the process may react with the metals of the reaction vessels, leading to their corrosion and passing traces of metal impurities into the solution, contaminating the final product.

Example- Acid like HCl if by chance contain a small amount of fluoride, it can itch the glass lining and begin the metallic contamination. Lead, antimony, bismuth etc. can crop up as impurities from the vessels.

Intermediate products in manufacturing process-

Some intermediate which are produced during the manufacture may be carried out through the final product as impurities. In the manufacturing process of potassium iodide, the intermediate iodate is the main impurity.

Manufacturing Hazards- In industrial areas, the atmosphere is contaminated with dust particle, silica glass, carbon gases. During the manufacture of pharmaceutical products, these impurities may enter the final products and alters the product potency.

2. During purification and processing— Often if not properly controlled, impurities also get added during the purification processes, mainly through the purifying reagents, solvents or vessels used.

Reagent used to remove other impurities- Sometime some chemicals are added to remove or to participate another substance. This may be also giving rise to source of impurity.

For example-

$BaCl_3$ is added to remove excess of sulphate in $AlCl_3$, hence $AlCl_3$

Water is the cheapest solvent and most widely used. Therefore, it is known as universal solvent.

Contamination due to vessels and equipment (filters, centrifuges, dryers etc) used for purification— During the purification processes, if the vessels are defective or not perfectly cleaned and dried, they may add impurities like metallic ions, rust, glass particle, moisture etc.

3. During storage and packing—

Errors in packaging materials-

During the process of packaging or filling and sealing, proper material which can ensure complete foolproof packaging without access to the atmosphere and light will ensure the stability of the product. Thus, quality and strength of packaging material is very important.

For example-

if the aluminium foil for the tablet strip or capsule for a liquid formulation bottle is of substandard quality it can add to impurities.

Faulty packaging process-

Most of the pharmaceutical packaging processes are assembly lined automated process, generally involving pressing and sealing with heat. If the process parameters are not optimized or tampered with, then it may lead to contaminations and can be hazards.

Microbial contamination- Microbial contamination, mainly in the form of fungal and bacterial growth may be due to the result of improper storage conditions as well as faulty packaging. The products for parenteral administration and ophthalmic preparations have to undergo sterility testing.

EFFECT OF IMPURITIES

• Impurities are sometime harmless, but are present more than certain limits then it lowered the active strength of the substance. The therapeutics effects of the drug also altered by the impurities.

• Impurities may bring about an incompatibility in the original substance and cause the deterioration in the substance.

• Some impurities take direct participation in the chemical reaction and change the chemical behaviour of the original substances.

• Impurities, even when present in traces, may show a cumulative toxic effect after a certain period.

• Some impurities promote the microbial growth and that are responsible for the deterioration of the substances.

• Some impurities may be able to catalyse the degradation, thereby shortening the shelf life of the drug substance.

• Some impurities by virtue of their unstable nature like hygroscopic nature, oxidisable nature etc. Can bring about change in the physical properties like change in appearance, taste, odour, stability etc. of drug substance causing technical difficulties in its use as well as formulation.

4. Write note on following.

i)primary and secondary standard.

ii) various technique of analysis.

STANDARDS

• Standards are very pure reagents.

• Their concentration are accurately known.

• We can express them with definite numbers and proper units. Uses Of standards

• to provide a reference using which we can determine the concentration of an unknown solution.

• Standardization of volumetric solutions

• To calibrate an instrument.

Types Of standards

• Primary standards

• Secondary standards

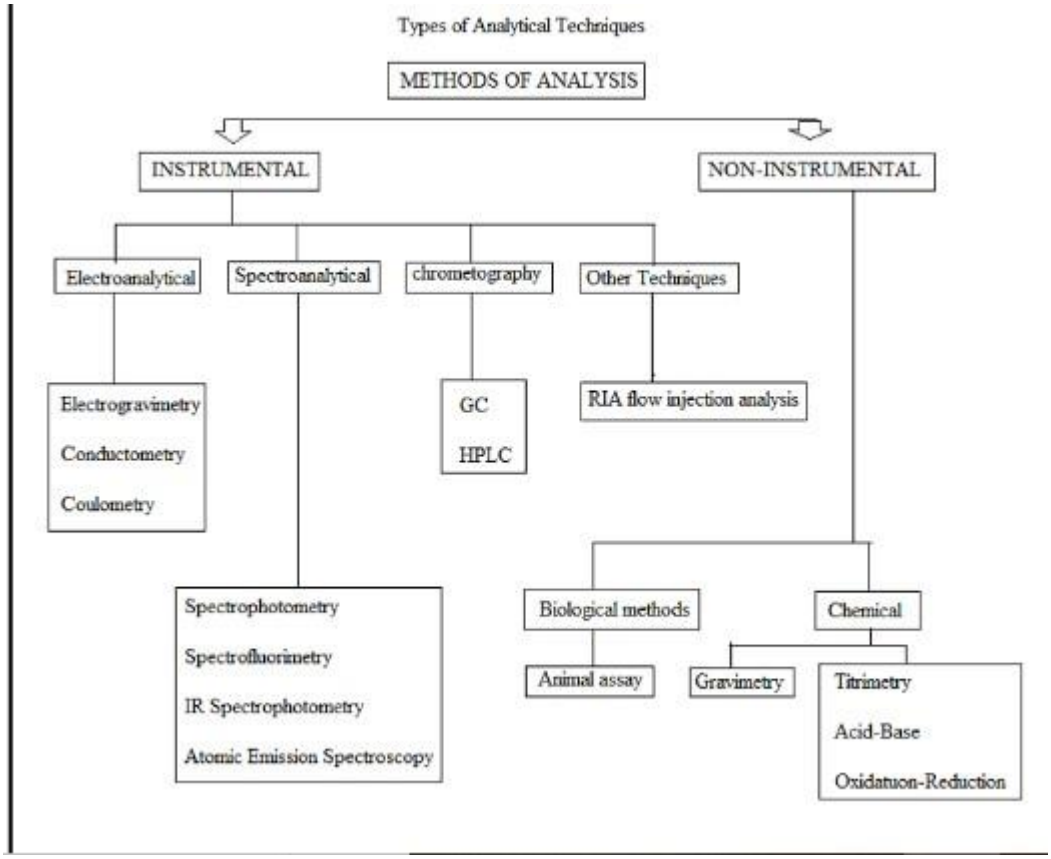
Primary Standards

Primary standards are reagents with accurately known concentration t o a known amount and very high purity which after dissolving of solvent gives primary standard solution'

Properties

- It should be 10% pure (0-01-0-02%. is tolerable)
- It should be stable at atmospheric condition.
- It must have high molecular and equivalent weight.
- It must have high stability and low reactivity.
- It should be non-hygroscopic and non- toxic.
- it must be inexpensive and readily available.

various technique of analysis.


Types of Analytical Techniques
METHODS OF ANALYSIS
INSTRUMENTAL
NON-INSTRUMENTAL
Electroanalytical
Spectroanalytical
chrometography
Other Techniques
Electrogravimetry
Conductometry
Coulometry
GC
HPLC
RIA flow injection analysis
Spectrophotometry
Spectrofluorimetry
IR Spectrophotometry
Atomic Emission Spectroscopy
Biological methods
Chemical
Animal assay
Gravimetry
Titrimetry
Acid-Base
Oxidation-Reduction

5. Write down the scope and method of analysis.

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6. Discuss the limit test for chloride and iron.

Limit test of chloride

Apparatus- Nessler's cylinder, pipette, stirring rod, beaker, stand.

Chemicals- Dilute nitric acid (10%) Silver nitrate (5%), test sample, standard sample (Sodium chloride).

Chemical structure

$$Cl^- + AgNO_3 \xrightarrow{\text{dil } HNO_3} AgCl\downarrow + NO_3^-$$

Principle— The limit test of chloride is based upon the chemical reaction between the soluble chloride ion with a silver nitrate reagent in a nitric acid media. The insoluble silver chloride renders the test solution turbid (depending upon the amount of silver chloride formed and therefore, on the amount of chloride present in the substance under test.

The turbidity is compared with the standard turbidity produced by the addition of silver nitrate, to the known amount of chloride ion (sodium chloride) solution. If the test solution shows less turbidity than the standard, the sample passes the test.

Procedure: Test solution

- Dissolve the test sample in water and transfer to the Nessler cylinder.
- Then add 1ml of dilute nitric acid and make the volume 50ml by adding water.
- Finally add 1ml of silver nitrate and stir immediately with stirring rod and set aside for 5 minutes. Observe the opalescence developed and compare with that of the test sample.

Standard solution

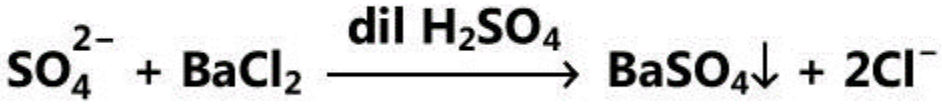
- 1ml of standard sample (0.05845% w/v) add in another cylinder.
- Then add the 10ml of nitric acid and make up the volume 50ml by adding water.
- Finally add 1ml of silver nitrate and stir immediately with stirring rod and set aside for 5 minutes. Observe the opalescence developed and compare with that of the standard sample.

Limit test for Sulphate.

Requirement:

Apparatus — Nessler's cylinder, pipette, stirring rod, beaker, stand.

Chemical reaction



Principle— In the limit test for sulphate, the solution of the substances undertest is mixed with barium chloride reagent in the presence of dilute hydrochloric acid then turbidity produced.

After this, perform standard experiment in similar manner with a known quantity of sulphate ion (using potassium sulphate) . The substance passes the limit test if it produces turbidity that is less than the standard. --- hydrochloric acid test sample, standard sample, barium chloride.

Limit Test for Iron.

Requirement:-

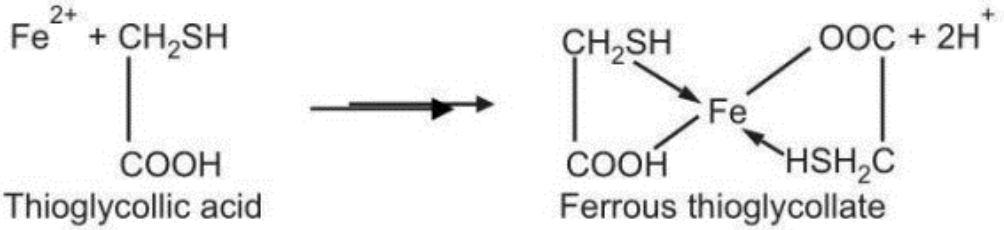
Apparatus — Nessler's cylinder, pipette, stirring rod, beaker, stand. Chemicals— Test sample, standard sample, iron-free citric acid, iron-free ammonia solution, thioglycolic acid.

Principle— This test is based upon the reaction of iron in an ammonia solution, with thioglycolic acid which forms a pink to deep reddish purple coloured complex of iron – thioglycolate.

Iron present in ferrous form and quite stable for long period in the absence of air. the colour are destroyed by oxidizing agent and strong alkali. The original state of iron is unimportant, as thioglycolic acid reduces fe2+ to fe3+ .

Then compared the test solution with standard solution (ferritic ammonium sulphate). It's the colour from test solution is less dark than the standard, then the sample passes the test.

Chemical structure



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Procedure

Test solution

• Test sample dissolved in water in Nessler cylinder and make up the volume 40 ml.

• Then add 2ml of 20%w/v solution of iron free citric acid and 0.1ml thioglycolic acid then mix.

• Make alkaline with iron free ammonia solution and make up volume 50ml.

• Observe the intensity of the purple colour developed by viewing vertically and compare with that of the test sample.

Standard solution

• Take 2ml of standard iron solution in Nessler cylinder and make up volume 40ml by adding water.

• Then add 2ml of 20%w/v solution of iron free citric acid and 0.1ml thioglycolic acid then mix.

• Make alkaline with iron free ammonia solution and make up volume 50ml.

• d. Observe the intensity of the purple colour developed by viewing vertically and compare with that of the standard sample.

Inference of other metal cation is eliminated by making use of 20% citric acid which forms complex with other metal ions. Earlier ammonium thiocyanate reagent was used for the limit test of iron. Since thioglycolic acid is more sensitive reagent for iron .it has replaced ammonium thiocyanate in the test.

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7. Write down the preparation and standardization of sodium thiosulphate.

Preparation of 0.1 N Sodium Thiosulfate Solution and Standardization:

1. Preparation:

- **Weighing:** Accurately weigh the required amount of sodium thiosulfate (Na2S2O3) using an analytical balance. The molar mass of sodium thiosulfate is 158.11 g/mol.
- **Dissolution:** Dissolve the weighed sodium thiosulfate in distilled water in a suitable container. Stir the solution thoroughly to ensure complete dissolution.
- **Volumetric Adjustment:** Transfer the solution to a volumetric flask of known volume, such as a 1000 mL flask. Rinse the weighing container with distilled water to ensure all the sodium thiosulfate is transferred to the flask. Then, fill the flask to the mark with distilled water, using a dropper or a funnel for precision.
- **Mixing:** Cap the flask and invert it several times to ensure homogeneity. This results in a 0.1 N sodium thiosulfate solution, where the normality is precisely defined by the molarity and the volume of the volumetric flask.

2. Standardization of Sodium Thiosulfate:

To standardize the sodium thiosulfate solution, perform a titration with a primary standard iodine solution.

- **Preparation of Iodine Solution:** Dissolve a known amount of potassium iodate (KIO3) in water, then add excess potassium iodide (KI) to convert it to iodine (I2).
- **Titration:** Titrate the prepared sodium thiosulfate solution against the iodine solution. Record the volume of sodium thiosulfate required to react completely with the iodine.
- **Calculation:** Use the stoichiometry of the reaction between sodium thiosulfate and iodine to calculate the exact normality of the sodium thiosulfate solution. The balanced chemical equation for the reaction is: $Na2S2O3+I2\rightarrow Na2S4O6+2NaI$.
- **Repeat:** Repeat the titration if necessary to ensure accuracy and precision.

importance

The preparation and standardization of the 0.1 N sodium thiosulfate solution are crucial in analytical chemistry. The accurate determination of normality ensures reliable and consistent results when this solution is used in various titrations, particularly in the quantitative analysis of substances that can react with iodine.

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In summary, the meticulous preparation and standardization of the 0.1 N sodium thiosulfate solution involve precise measurements, dissolution, volumetric adjustments, and subsequent titration with a primary standard, ensuring the accuracy of the solution's normality for analytical applications.

Short question (5marks)

1. write down the sources of error.

Sources of systematic Errors—

sources are mentions below.

Instrumental Errors— Due to the use of defective equipment or low-quality instruments, errors are arising in the analytical procedure. It is easily checkable by the analyst.

Proportional Errors— The absolute value of this kind of the errors changes with the size of the sample in such a fashion that the relative error remains constant. It is easily incorporated by a material that directly interferes in analytical process.

Personal Errors— Errors are induced due to the carelessness, or ignorance and lack of skilled. This error is also called operative error. It is occurring by who are handling the method of analysis.

Chemical/Reagent Errors— Chemical errors are based on the chemical reactivity between the using chemical and reagent.

Errors in Methodology— It is a most serious error in analysis, as the error arises due to faulty methods.

2. Indeterminate errors-

Indeterminate or Random Errors— In this error we not define the specific well-known reason and cannot be eliminated, so it is also called as accidental errors.

• It is induced by the several successive measurements performed by the same analyst under the same conditions and identical experiments.

Such accidental errors will follow a random distribution pattern and the mathematical law of probability can be applied to get net conclusion regarding the results.

2. What is the scope of pharmaceutical analysis.

The scope of pharmaceutical analysis refers to the range of activities and areas in which analytical techniques and methods are employed in the pharmaceutical industry to ensure the quality, safety, and efficacy of pharmaceutical products. Here are some key points that outline the scope of pharmaceutical analysis:

1. **Drug Development**: Pharmaceutical analysis is integral to drug development, where it is used to characterize and quantify the active pharmaceutical ingredient (API), as well as to assess the purity and stability of drug candidates.
2. **Quality Control (QC)**: It plays a crucial role in QC, ensuring that manufactured pharmaceutical products meet predefined quality standards. This includes the analysis of raw materials, in-process materials, and finished products.
3. **Pharmacopoeia Compliance**: Pharmaceutical analysis is used to ensure that products adhere to the standards set by pharmacopoeias, such as the United States Pharmacopeia (USP) and the European Pharmacopoeia (Ph. Eur).
4. **Stability Testing**: Analytical techniques are employed to assess the stability of pharmaceutical products under various storage conditions, which is crucial for establishing shelf life and ensuring product quality.
5. **Regulatory Compliance**: To meet regulatory requirements, pharmaceutical companies must perform extensive analysis to demonstrate the safety and efficacy of their products, as mandated by regulatory agencies like the FDA (Food and Drug Administration).
6. **Bioequivalence Studies**: For generic drug manufacturers, pharmaceutical analysis is essential to demonstrate bioequivalence to the reference innovator product, often through in vitro and in vivo studies.
7. **Dissolution Testing**: Analytical methods, such as dissolution testing, are used to evaluate the rate at which a drug dissolves in the body, which is important for drug delivery and formulation design.
8. **Analytical Method Development**: Developing and validating new analytical methods for specific drugs or formulations to improve sensitivity, accuracy, and efficiency.

9. **Trace Analysis**: Detecting and quantifying impurities and trace substances in pharmaceutical products, which can impact safety and efficacy.
10. **Pharmaceutical Biotechnology**: Analysing biopharmaceuticals, such as monoclonal antibodies, peptides, and gene therapies, to ensure their safety and effectiveness.
11. **Pharmaceutical Microbiology**: Assessing microbial contamination and sterility in pharmaceutical products, especially in sterile formulations.

12. **Pharmaceutical Packaging Analysis**: Evaluating the integrity of packaging materials to ensure they protect pharmaceutical products from external factors.
13. **Pharmacokinetics and Pharmacodynamics**: Analysing the concentrations of drugs and their metabolites in biological matrices to understand their absorption, distribution, metabolism, and elimination (ADME).

14. **Pharmaceutical Research**: Analytical techniques are used in research to investigate the physicochemical properties of drugs and excipients, as well as their interactions.
15. **Counterfeit Drug Detection**: Employing analysis to identify counterfeit and substandard drugs in the market to protect public health.
16. **Environmental Monitoring**: Monitoring and controlling the release of pharmaceutical compounds and byproducts into the environment to minimize environmental impact.
17. **Post-Marketing Surveillance**: Continuously analysing marketed pharmaceutical products to ensure ongoing quality and safety, as well as to detect and address any emerging issues.
18. **Personalized Medicine**: Analytical methods are used to tailor drug therapies to individual patients based on their genetic and physiological characteristics.

3. Explain the principle involved in limit test for iron why citric acid and ammonia solution are added into it.

Limit Test for Iron.

Requirement:-

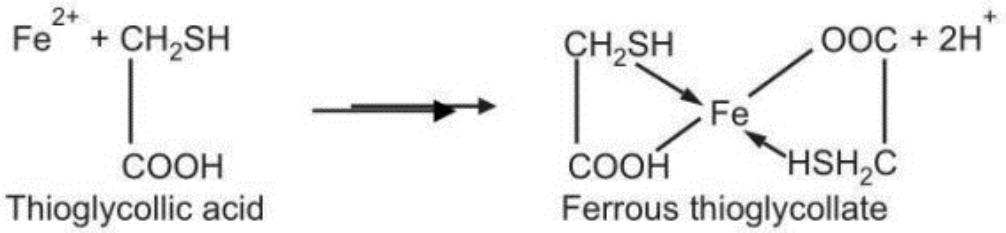
Apparatus — Nessler's cylinder, pipette, stirring rod, beaker, stand. Chemicals— Test sample, standard sample, iron-free citric acid, iron-free ammonia solution, thioglycolic acid.

Principle— This test is based upon the reaction of iron in an ammonia solution, with thioglycolic acid which forms a pink to deep reddish purple coloured complex of iron – thioglycolate.

Iron present in ferrous form and quite stable for long period in the absence of air. the colour are destroyed by oxidizing agent and strong alkali. The original state of iron is unimportant, as thioglycolic acid reduces fe2+ to fe3+ .

Then compared the test solution with standard solution (ferritic ammonium sulphate). It's the colour from test solution is less dark than the standard, then the sample passes the test.

Chemical structure



Procedure

Test solution

• Test sample dissolved in water in Nessler cylinder and make up the volume 40 ml.

• Then add 2ml of 20%w/v solution of iron free citric acid and 0.1ml thioglycolic acid then mix.

• Make alkaline with iron free ammonia solution and make up volume 50ml.

• Observe the intensity of the purple colour developed by viewing vertically and compare with that of the test sample.

Standard solution

• Take 2ml of standard iron solution in Nessler cylinder and make up volume 40ml by adding water.

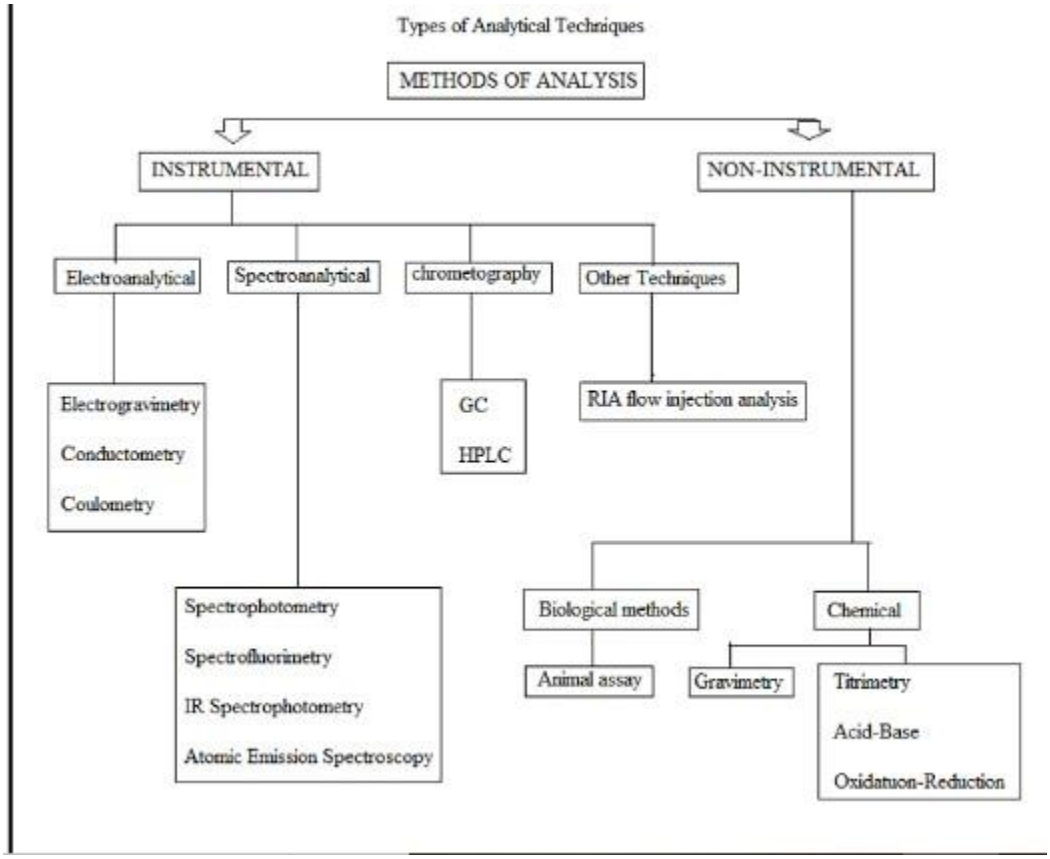
• Then add 2ml of 20%w/v solution of iron free citric acid and 0.1ml thioglycolic acid then mix.

• Make alkaline with iron free ammonia solution and make up volume 50ml.

• d. Observe the intensity of the purple colour developed by viewing vertically and compare with that of the standard sample.

Inference of other metal cation is eliminated by making use of 20% citric acid which forms complex with other metal ions. Earlier ammonium thiocyanate reagent was used for the limit test of iron. Since thioglycolic acid is more sensitive reagent for iron .it has replaced ammonium thiocyanate in the test.

4. What are the various technique of analysis.



5. Discuss the limit test for lead using dithizone.

Limit test for heavy metal.

Requirement:

Apparatus— Nessler's cylinder, pipette, stirring rod, beaker, stand. Chemicals—Test sample, standard sample, dilute acetic acid, dilute ammonia, dilute sodium hydroxide hydrogen sulphide solution. Chemical reactions-----

Principle-

The limit test for heavy metal is based upon the reaction of the metal ion with hydrogen sulphide, under the prescribed conditions of the test, resulting in the formation of metal sulphides. These remains distributed in a colloidal state and produce a brownish coloration.

The heavy metal is the metallic inclusion that are darkened with sodium sulphide (TS) in acidic solution or hydrogen sulphide saturated solution as their quantity is expressed in terms of the quantity of lead (Pb).

The metallic impurities in substances are expressed as parts of lead per million parts of the substances. The usual limits as per I.P is 20ppm.

Chemical structure

$$Pb(NO_3)_2(Pb^+) + H_2S \rightarrow PbS\downarrow + 2HNO_3$$

$$PbCl_2 + Na_2S \rightarrow PbS\downarrow + 2NaCl$$

Procedure---

Test solution

- Take the test sample and 20ml water maintain in Nessler cylinder. Then add 5ml of dilute sodium hydroxide and make up the volume up to 50ml Finally add the 5 drops of sodium sulphide solution and stir well and set aside for 5 minutes. Observe the darkness of colour and compare with that of the standard.

Standard solution

- Take the 2ml of standard solution in 20ml of water in Nessler cylinder.
- Then add 5ml of dilute sodium hydroxide and make up the volume up to 50ml
- Finally add the 5 drops of sodium sulphide solution and stir well and set aside for 5 minutes.
- Observe the darkness of colour and compare with that of the standard.

6. Discuss about gutziet apparatus.

Apparatus:

An apparatus as per the specification of I P is used for the limit test for arsenic.

A wide mouth bottle of 120 ml capacity fitted with rubber bung carrying a glass of tube 200 mm long and 6.5 mm internal E diameter with a hole of 2 mm at one end is a used in the test.

The other end of the glass tube is cut smooth carries rubber bungs (25 x 25 mm). Mercuric chloride paper is sandwiched between the rubber bungs. The rubber bungs are held in place by means of a clip.

7. Write down the preparation of sodium hydroxide and sulphuric acid.

1. Preparation of 0.1 M Sulfuric Acid:

- **Weighing:** Accurately weigh the required amount of sulfuric acid (H_2SO_4) using an analytical balance. The molar mass of sulfuric acid is 98.08 g/mol.
- **Dilution:** Since concentrated sulfuric acid is typically available in higher concentrations, you will need to dilute it to achieve the desired molarity. Use the formula $C_1V_1=C_2V_2$, where C_1 is the concentration of the concentrated solution, V_1 is the volume to be taken
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- from the concentrated solution, C_2 is the desired concentration (0.1 M), and V_2 is the total volume after dilution.
- **Volumetric Adjustment:** Transfer the diluted sulfuric acid solution to a volumetric flask of known volume, such as a 1000 mL flask. Rinse the dilution container with distilled water to transfer any remaining acid.

Mixing: Cap the flask and invert it several times to ensure thorough mixing. This results in a 0.1 M sulfuric acid solution **Preparation of 0.1 N Sodium Hydroxide Solution and Standardization:**

1. Preparation:

To prepare a 0.1 N (Normal) solution of sodium hydroxide (NaOH), follow these steps:

- **Weighing:** Accurately weigh the required amount of sodium hydroxide pellets or flakes. The molar mass of NaOH is 39.997 g/mol.

- **Dissolution:** Dissolve the weighed sodium hydroxide in distilled water in a suitable container. Stir the solution thoroughly to ensure complete dissolution.

- **Volumetric Adjustment:** Transfer the solution to a volumetric flask of known volume, such as a 1000 mL flask. Rinse the weighing container with distilled water to ensure all

- the sodium hydroxide is transferred to the flask. Then, fill the flask to the mark with distilled water, using a dropper or a funnel for precision.

- **Mixing:** Cap the flask and invert it several times to ensure homogeneity. This results in a 0.1 N sodium hydroxide solution, where the concentration is precisely defined by the normality and the volume of the volumetric flask.

8. Difference between primary and secondary standard.

STANDARDS

• Standards are very pure reagents.

• Their concentration are accurately known.

• We can express them with definite numbers and proper units. Uses Of standards

• to provide a reference using which we can determine the concentration of an unknown solution.

• Standardization of volumetric solutions

• To calibrate an instrument.

Types Of standards

• Primary standards

• Secondary standards

Primary Standards

Primary standards are reagents with accurately known concentration t o a known amount and very high purity which after dissolving of solvent gives primary standard solution'

Properties

- It should be 10% pure (0-01-0-02%. is tolerable)
- It should be stable at atmospheric condition.

- It must have high molecular and equivalent weight.
- It must have high stability and low reactivity.
- It should be non-hygroscopic and non- toxic.

- it must be inexpensive and readily available.

Short question. (2 marks)

- Difference between accuracy and precision.

Accuracy— Nearest or accurate value which are matches to the true value of any experiments is defined the term accuracy.

Precisions.

Precisions are defined as the agreement amongst a cluster of experimental results, however, it does not imply anything with respect to their relation to the 'true value' precisions designates 'reproducibility' of a measurement, where accuracy, but ironically a high degree of precision may not necessarily suggest accuracy.

2. Define pharmaceutical analysis.

The pharmaceutical analysis is a branch of chemistry, which involves the series of process for the identification, determination, quantitation, and purification. This is mainly used for the separation of the components from the mixture and for the determination of the structure of the compounds.

- Define error.

Errors— Errors is defined as the deformity present in any measurements by addition of any internal or external factor.

In the pharmaceutical science errors are induced by the defective equipment and methods.

In analytical chemistry errors are affects the material products reliability, reproducibility, and accuracy or precision.

- Why citric acid and ammonia is used in limit test for iron.

To maintain the balance in limit test for iron to 3-4 PH citric acid and ammonia is used.

- What is impurities.

Impurities is defined as the presence of undesired/unexpected material during any procedure and may alters the final products. The substances that are used in the pharmaceuticals should be pure enough to be used safely but it is difficult to obtain an absolute pure substance

- Define significant figure

  Significant figures (also known as the significant digits) of a number in positional notation are digits in the number that are reliable and absolutely necessary to indicate the quantity of something.

- Write down the chemical structure and basic principle of limit for chloride.

  Principle— The limit test of chloride is based upon the chemical reaction between the soluble chloride ion with a silver nitrate reagent in a nitric acid media. The insoluble silver chloride renders the test solution turbid (depending upon the amount of silver chloride formed and therefore, on the amount of chloride present in the substance under test.

$$Cl^- + AgNO_3 \xrightarrow{\text{dil } HNO_3} AgCl\downarrow + NO_3^-$$

- Define the term accuracy.

  Accuracy— Nearest or accurate value which are matches to the true value of any experiments is defined the term accuracy

- Why potassium sulphate is added in limit test for sulphate.

  To increase the sensitivity of the reaction.

9. Define standards.

- Standards are very pure reagents. Their concentration are accurately known.

We can express them with definite numbers and proper units.

1. Write down the principle and chemical structure of limit test for lead.

   Principle-

   The limit test for heavy metal is based upon the reaction of the metal ion with hydrogen sulphide, under the prescribed conditions of the test, resulting in the formation of metal sulphides. These remains distributed in a colloidal state and produce a brownish coloration.

$$Pb(NO_3)_2(Pb^+) + H_2S \rightarrow PbS\downarrow + 2HNO_3$$

$$PbCl_2 + Na_2S \rightarrow PbS\downarrow + 2NaCl$$

11. Why phenol red is used in lit test for lead.

Phenol red is used as indicator in limit test for lead.

12. Define the term limit test.

Limit Test:

• Limit test is defined as quantitative or semi- quantitative tests which are performed to identify and control small amount of impurities which are likely to be present with the substance to be analysed. • For to substance clarification and purity limit test perform a key role in the pharmaceutical analysis. Generally, limit tests are carried out to identify the inorganic impurities present in the substance.

• Limit tests are not based on the numerical value.

13. Give 3 example of primary standards.

Sodim hydroxide, sulphuric acid, hydrochloric acid.

14. Why KI is used in limit test for arsenic.

KI is used as reducing agent in limit test for arsenic.

14. What is the principle of limit test for arsenic.

Principle—

The pharmacopoeia method is based on the Gutzeit test. In this test, arsenic gas released which when passed over a mercuric chloride test paper, produces a yellow stain. The intensity of the stain is proportional to the amount of arsenic presents. The rate of evolution of gas is maintained by using a particular size of zinc, and any impurities coming along with the gas is trapped by placing a lead acetate-soaked cotton plug in the apparatus.

MODEL QUESTION AND ANSWER

(10marks)

1) Write about the theories of acid base indicator.

Acid- Base indicator

An acid-base indicator is a substance that undergoes a visible change in colour or some other easily detectable property when it comes into contact with acidic or basic solutions. Indicators are often used in chemistry to determine the endpoint of a titration or to indicate the pH of a solution. The colour change is a result of the indicator's sensitivity to the concentration of hydrogen ions (H^+) or hydroxide ions (OH^-) in the solution.

Theory of acid base indicator

There are 2 theory of indicator theory are listed following.

Ostwald theory

2) Quinonoid theory

i) Ostwald's theory

Acid (A): Methyl Orange

Acid Ionization:
Methyl Orange (MO) is an acid-base indicator. In its acidic form, it can be represented as HMO^+.
In an acidic solution, it ionizes to produce hydrogen ions (H^+) and the conjugate base of the indicator (MO^-).
Acid Ionization: $HMO^+ \rightarrow H^+ + MO^-$

Base (B): Phenolphthalein

Base Ionization:
Phenolphthalein (PH) is another acid-base indicator. In its basic form, it can be represented as PH^-.
In a basic solution, it ionizes to produce hydroxide ions (OH^-) and the conjugate acid of the indicator (H_2PH).
Base Ionization: $PH^- + OH^- \rightarrow H_2PH + O^-$

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Salt Formation:

In a reaction between the hydrogen ions (H^+) from the acid (Methyl Orange) and the hydroxide ions (OH^-) from the base (Phenolphthalein), water (H_2O) is formed. Remaining ions combine to form a salt, but since these are indicators, salts are not typically considered in the context of Ostwald's theory.

Overall Reaction:

The overall reaction between the acid (Methyl Orange) and the base (Phenolphthalein) can be represented as:
$HMO^+ + PH^- + OH^- \rightarrow H_2O + MO^- + H_2PH + O^-$

This example illustrates how Ostwald's theory applies to the ionization of two acid-base indicators, Methyl Orange and Phenolphthalein, in the presence of a base. The hydrogen ions from Methyl Orange react with the hydroxide ions from Phenolphthalein, forming water and the conjugate bases of the indicators. Ostwald's theory provides a basic understanding of the ionization processes in acidic and basic solutions.

ii) Quinonoid theory

Quinonoid compounds typically involve a quinone structure, which is a six-membered carbon ring with alternating double bonds. These compounds often exhibit colour changes based on their electronic configuration, making them suitable for use as indicators.

Now, let's discuss Methyl Orange and Phenolphthalein:

Acid (A): Methyl Orange

Quinonoid Structure:
Methyl Orange (MO) contains a quinonoid structure in its molecular arrangement. The quinonoid structure contributes to the colour changes observed during acid-base titrations.

Base (B): Phenolphthalein

Quinonoid Structure:
Phenolphthalein (PH) does not have a typical quinonoid structure.
Phenolphthalein, however, undergoes a colour change during titrations, transitioning from colorless to pink in a basic environment.

Acid-Base Interaction:

Acid Ionization (Methyl Orange):
Methyl Orange in its acidic form (HMO^+) undergoes ionization in solution.
Acid Ionization: $HMO^+ \rightarrow H^+ + MO^-$

$\rightarrow H^+ + MO^- + PH^- + H_2O$

This representation combines the ionization of Methyl Orange in an acidic solution and the interaction of Phenolphthalein with hydroxide ions in a basic solution. While the terminology may not align perfectly with a specific "Quinonoid theory," it aims to illustrate the acid-base characteristics of these indicators with a focus on their molecular structures and color changes during chemical interactions.

Limitation of Lewis theory

Neglects Molecular Geometry: Lewis's theory does not provide information about the three-dimensional shape of molecules. The arrangement of atoms in space influences the properties and reactivity of molecules, which Lewis structures alone do not account for.
Lacks Explanation for Molecular Orbital Theory: Lewis's theory does not consider molecular orbitals, which are essential for understanding the electronic structure of molecules. Molecular orbital theory provides a more accurate depiction of bonding and electron distribution compared to the simplistic electron-pair sharing model of Lewis structures.
Inadequate Treatment of Resonance: Lewis structures imply a fixed arrangement of electrons within a molecule, ignoring situations where electrons are delocalized and can exist in multiple locations. Resonance structures are used to address this, but Lewis's theory itself does not explicitly incorporate the concept of resonance.

2) Define various neutralization curve for acid base titration.

Neutralization curve

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A neutralization curve is a graphical representation of the pH changes that occur during a titration of an acid with a base or vice versa. The curve typically displays the pH of the solution being titrated as a function of the volume of the titrant (the solution being added). The pH of the solution changes as the titration progresses due to the neutralization reaction between the acid and the base.

Different neutralization curve for different acid- base titration.

Strong acid and strong base

Weak acid and strong base

Strong acid and weak base

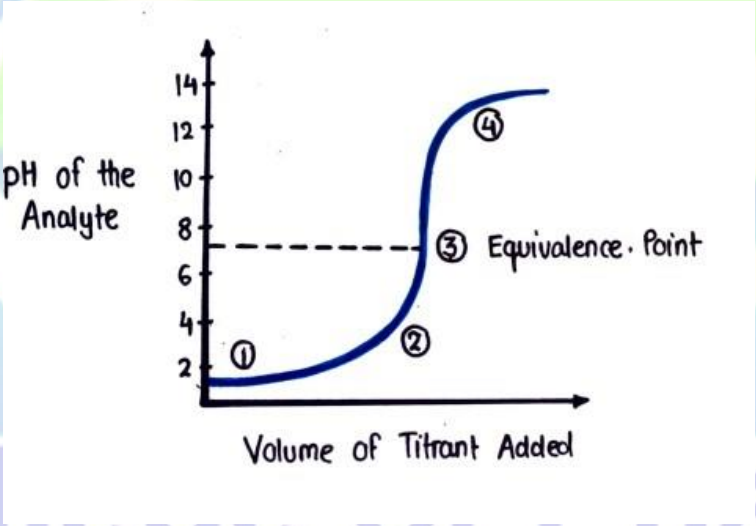
Weak acid and weak base

Neutralization curve of strong acid and strong base

A neutralization curve illustrates the pH changes that occur during the titration of an acid with a base. The shape of the curve depends on the strength of the acid and the base involved in the reaction. Let's discuss the neutralization curves for the four scenarios mentioned:

Strong Acid and Strong Base:

Neutralization Reaction: $HCl(aq)+NaOH(aq)\rightarrow NaCl(aq)+H_2O(l)$

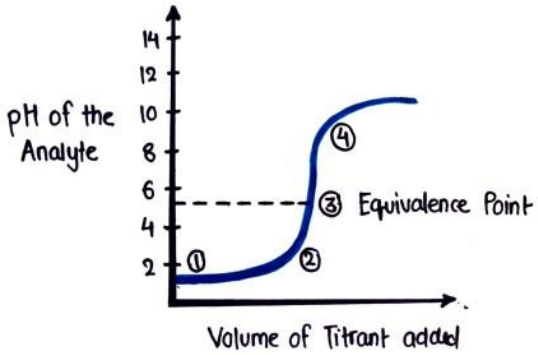


Explanation: In this case, both the acid (HCl) and the base (NaOH) are strong. The pH starts very low (acidic) and gradually increases as the base is added. The equivalence point occurs around pH 7, indicating a neutral solution.

Weak Acid and Strong Base:

Neutralization Reaction:

$$CH_3COOH(aq)+NaOH(aq)\rightarrow CH_3COONa(aq)+H_2O(l)$$

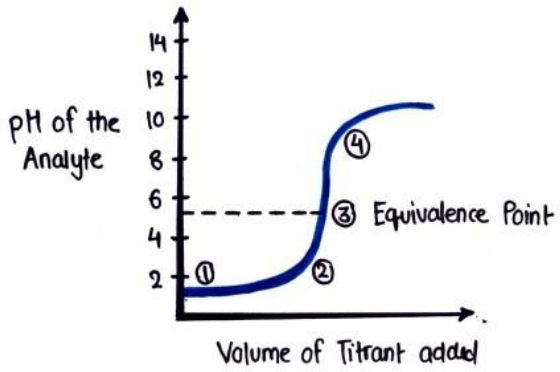


Explanation: The weak acid (CH_3COOH) results in a slower increase in pH compared to a strong acid. The pH at the equivalence point is greater than 7, indicating a slightly basic solution.

Strong Acid and Weak Base:

Neutralization Reaction:

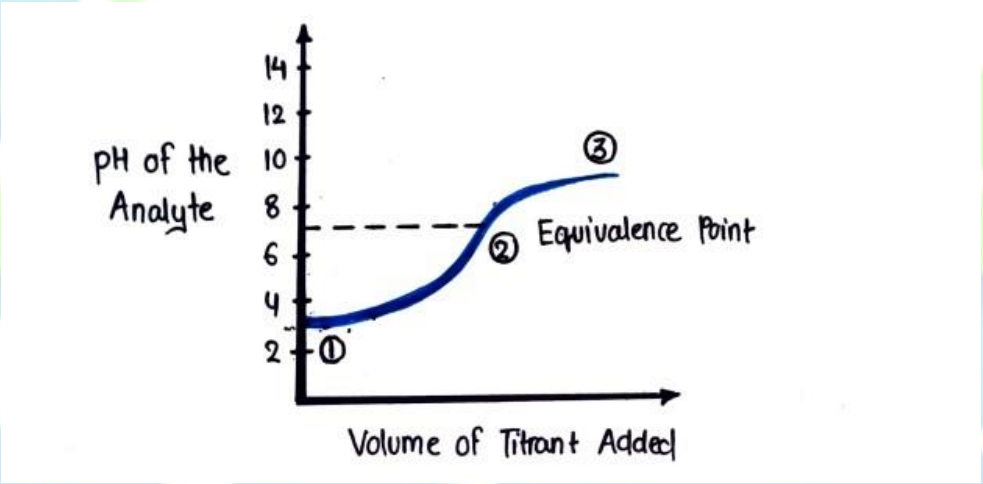
$$HCl(aq)+NH_3(aq)\rightarrow NH_4Cl(aq)$$



Explanation: The weak base (NH_3) leads to a slower rise in pH. The equivalence point in this case is less than 7, indicating a slightly acidic solution.

Weak Acid and Weak Base:

Neutralization Reaction: $CH_3COOH(aq)+NH_3(aq)\rightarrow CH_3COONH_4(aq)$



Explanation: Both the weak acid and weak base result in a curve with a gradual increase in pH. The equivalence point is typically around pH 7, indicating a neutral or nearly neutral solution.

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3) Write about the theories of acid base titration.

There are three theories of acid and base

Arrhenius theory

Bronsted-Lowry theory

Lewis's theory

Arrhenius theory

Proposed by Svante Arrhenius in 1884

Definition of acid

According to the Arrhenius theory, an acid is defined as a substance that, when dissolved in water, increases the concentration of hydrogen ions (H +) in the aqueous solution.

In other words, acids are substances that ionize to produce protons (hydrogen ions) when dissolved in water.

It is also known as neutralization reaction.

For example,

when hydrogen chloride (HCl) is dissolved in water, it ionizes to produce hydrogen ions and chloride ions:

$$HCl \rightarrow H^+ + +Cl -$$

Definition of base

According to the Arrhenius theory, a simple definition of a base is that it is a substance that, when dissolved in water, increases the concentration of hydroxide ions ($OH–$) in the aqueous solution.

Bases, in the Arrhenius sense, are substances that ionize to produce hydroxide ions when mixed with water.

Example

When sodium hydroxide ($NaOH$) is dissolved in water, it ionizes to produce hydroxide ions:

$$NaOH \rightarrow Na^+ + OH^-$$

In this reaction, $NaOH$ acts as a base according to the Arrhenius definition because it increases the concentration of OH^- ions in the solution.

Limitation of Arrhenius theory

Limited to solution

The theory is only applicable to substances that ionize in water.

Example:

Acids and bases that do not dissolve or ionize in water are not explained by the Arrhenius theory.

Exclude atmospheric substance

The theory does not account for substances that can act as both acids and bases.

Example:

Water (H_2O), which can act as both an acid and a base in different circumstances.

No explanation about gaseous acid and base

: The theory does not address the acidic or basic nature of gases.

Example

Hydrogen chloride gas (HCl) is acidic, but this is not explained by the Arrhenius theory.

Limited to proton transfer

The theory only considers the transfer of protons(H+).

Example:

The reaction between ammonia (NH_3) and boron trifluoride (BF_3), which involves the donation of a pair of electrons, is not explained.

Bronsted-Lowry theory

Acid

Definition:

A Brønsted-Lowry acid is a substance that donates a proton (H^+ ion) to another substance.

example

$$HCl + H_2O \rightarrow Cl^- + H_3O$$

In the reaction **HCl** acts as a Brønsted-Lowry acid because it donates a proton to. H_2O

Base

Definition:
A Brønsted-Lowry base is a substance that accepts a proton (H^+ ion) from another substance.
example

$$NH_3 + H_2O \rightarrow NH_4^+ + OH^-$$

In the reaction NH_3 acts as a Brønsted-Lowry base because it accepts a proton from H_2O.

CONJUGATE ACID-BASE PAIR

CONCEPT –
In any acid-base reaction, there are conjugate acid-base pairs where the acid of one pair is related to the base of another by the transfer of a proton.
EXAMPLE
In the equilibrium reaction $H_2O + NH_3 \rightleftharpoons H_3O^+ + NH_2$, H_2O and H_3O^+ form a

conjugate acid-base pair, as do NH_3 and NH_2

The conjugate acid-base pair associated with water is represented as:

$H2O \rightleftharpoons H^+ + OH^-$

In this equilibrium, water can function as both an acid ($H_2O \rightarrow H^+ + OH^-$) and a base ($H^+ + OH^- \rightarrow H_2O$), highlighting the amphiprotic nature of the conjugate acid-base

Lewis's theory

The Lewis acid-base theory, proposed by American chemist Gilbert N. Lewis in 1923, defines acids and bases based on the transfer of electron pairs. According to this theory:

Lewis Acid:

A Lewis acid is a substance that can accept a pair of electrons.

In Lewis acid-base interactions, the Lewis acid is often a species with an electron-deficient center, typically a metal cation or a molecule with an incomplete octet of electrons.

Lewis Base:

A Lewis base is a substance that can donate a pair of electrons.

In Lewis acid-base interactions, the Lewis base is typically a species with a lone pair of electrons. This lone pair is donated to the Lewis acid.

Lewis Acid-Base Interaction:

Lewis's acid-base interactions involve the sharing of electron pairs, where the Lewis acid accepts electrons from the Lewis base.

The resulting bond is a coordinate covalent bond, where both electrons in the shared pair come from the Lewis base.

Examples:

One of the classic examples of Lewis acid-base interaction is the reaction between boron trifluoride (BF_3), which acts as a Lewis acid, and ammonia (NH_3), acting as a Lewis bas

$$BF_3 + NH_3 \rightarrow BF_3NH_3$$

In this reaction, the nitrogen lone pair in NH_3 donates to the electron-deficient boron in BF_3, forming a coordinate covalent bond.

Broader Applicability:

The Lewis acid-base theory is more inclusive than the Bronsted-Lowry theory, as it doesn't rely on the transfer of protons. It can explain a wider range of reactions, including those involving compounds without hydrogen.

Complex Formation:

Lewis's acid-base theory is particularly useful in describing the formation of coordination complexes in transition metal chemistry, where metal cations act as Lewis's acids by accepting electron pairs from ligands.

The Lewis acid-base theory provides a comprehensive framework for understanding chemical interactions involving electron-pair transfer, offering insights into a variety of reactions beyond traditional acid-base reactions.

Limitation of Lewis theory

Neglects Molecular Geometry: Lewis's theory does not provide information about the three-dimensional shape of molecules. The arrangement of atoms in space influences the properties and reactivity of molecules, which Lewis structures alone do not account for.
Lacks Explanation for Molecular Orbital Theory: Lewis's theory does not consider molecular orbitals, which are essential for understanding the electronic structure of molecules. Molecular orbital theory provides a more accurate depiction of bonding and electron distribution compared to the simplistic electron-pair sharing model of Lewis structures.
Inadequate Treatment of Resonance: Lewis structures imply a fixed arrangement of electrons within a molecule, ignoring situations where electrons are delocalized and can exist in multiple locations. Resonance structures are used to address this, but Lewis's theory itself does not explicitly incorporate the concept of resonance.

(5marks)

1)What is Non aqueous titration, define it advantage and disadvantage.

Non-aquas titration

Most of the titrations are performed in the aqueous media, means water is used as solvent, but in the case of weak acids and weak

bases, use of water don't give a sharp end

• Non- Aqueous solvents are those which do not contain waters.

• Non- Aqueous Titration refers to a type of titration in which the

analyte is dissolved in a solvent which doesn't contain water.

Reason For Non- Aqueous Titration

• The substance is insoluble in water. • The substance is reactive with water

• The sample is too weak acid or too weak base.

Advantages - Non-Aqueous Titration

• Organic acids or bases that are insoluble in water are easily soluble in non-aqueous solvent.

• It can be used for titration of mixture of acids as well.

• They are used for titration of weak acids and weak bases.

• Non- Aqueous Titrations are simple and accurate

Disadvantages of Non- Aqueous Titrations

• Non- Aqueous solvents are generally expensive.

• Volatile solvent can pollute environment.

• Indicator must be prepared in non-aqueous medium.

2)Discuss the various types of solvents used in non-aqueous titration.

Types OF Non-Aqueous solvents

There are basically 4 types of solvent used in the O Protophilic Solvents

Protophilic Solvents

Proteogenic Solvents

Amphoteric Solvents

Aprotic Solvents

• The word protophilic stands for proton lover'

• Protophilic solvents are basic in nature.

• They are used to dissolve acidic analytes

• They possess a high affinity for proton

• examples: Pyridine, amine etc.

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Protogenic Solvents

• The word protogenic stands for "Proton Generator'

• Protogenic solvents are acidic in nature f they can donate proton.

• They are used to dissolve basic analytes.

• They have dielectric constant.

• examples: Glacial acetic acid, Formic acid etc.

Amphiprotic Solvents

• They work as both protogenic and protophilic solvent.

• These solvents behave as acid as well as b a s e

• Amphiprotic solvents can either accept or donate the proton

• examples: Alcohols, Methanol, Ethanol etc.

Aprotic Solvents

• These solvents are chemically inert.

• They are neither acidic or nor basic

• They do not accept or donate protons.

• They have low dielectric constant.

• examples: Benzene, Chloroform etc.

N O T e: The principle of Non-Aqueous Titration is based on Bronsted - Lowry Theory of acid and base.

Most Commonly Used Non- Aqueous Solvent

• Glacial acetic acid

• Acetonitrile

• Alcohols

• Dioxane

• Dimethyl formamide

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3)Write down estimation of sodium benzoate.

ASSAY OF SODIUM BENZOATE

Molecular Formula: $C_7H_s\ NaO_2$,

Molecular Weight: 144.1 g/mol

Structure

Sodium benzoate contains not less than 99% and not more than 100-5 %. of $C_7H_s\ NaO_2$.

Principle

$C_6H_5COONa + HClO_4 \rightarrow C_6H_5COOH + NaClo_4$

(Sodium benzoate) (Perchloric acid) (Benzoic acid) (Sodium Perchlorate)

Properties

• tI is a white, crystalline or granular powder

• It is odorless.

• It is hygroscopic in nature.

Preparation and standardization of 0-1 N Perchloric acid

• Weight about 0.25 g of sodium benzoate and dissolve in 20 ml of anhydrous glacial acetic acid.

• I f necessary then warm to 50^0 c and then cool

• Ad crystal violet or -1 Naptholbenzein as indicator.

• Titrate with 01 M perchloric acid till colour changes to green.

Calculation

• 0.01441 C_6H_5COONa = 1 ml of O.1N $\underline{HClO_4}$

%. Purity Of CEMSCONG = $\frac{\text{Vol of } HClO_4 \text{ X NFO HC104 X } 0.1441}{\text{Weight of } C_6H_5COONa \times 0.1}$

Uses

• Preservatives

• Additives etc.

4)Write a note acidimetry and alkalimetry.

ACIDIMETRY

• Acidimetry used to determine the concentration of base substances Using standard acid solution.

• In acidimetry, a known volume of a base is put into a conical flask, the solution is then titrated against a standard solution of acid taken in burette till equivalent point comes.

• Equivalent paint is the point at which no. of moles of analyte (base) is equal to the no. of moles of titrant (acid).

• Here in acidimetry

Acid: Used as Titrant taken in burette

Base: Used as Analyte taken in flask

• in acidimetry, acid is the standard solution.

Alkalimetry

• Alkalimetry used to determine the concentration of acid substances using standard base.

• In alkalimetry a known volume of an acid is put into a conical flask and then titrated against standard solution of base taken in burette.

• Equivalent point is the paint at which no of moles of analyte (acid) is equal to the number of moles of titrant (base)

• Here in

Alkalimetry

© Acid: Used as analyte taken in flask

© Base: Used as titrant taken in burete

• In, Alkalimetry, Base is the standard solution.

(2marks)

Define levelling effect.

• Levelling effect refers to the effect of solvents on the properties of acids and bases. It refers to increasing dissociation of compounds.
• Since al strong acids and bases are completely dissociates in water hence water has Levelling effect on strong acids and bases.
• But as we know weak acids and weak bases are hardly dissociates in water, hence water doesn't have a levelling effect no weak acids and weak bases

Define Lewis acid and base.

Lewis Acid:
A Lewis acid is a substance that can accept a pair of electrons.
In Lewis acid-base interactions, the Lewis acid is often a species with an electron-deficient centre, typically a metal cation or a molecule with an incomplete octet of electrons.
Lewis Base:
A Lewis base is a substance that can donate a pair of electrons.
In Lewis acid-base interactions, the Lewis base is typically a species with a lone pair of electrons. This lone pair is donated to the Lewis acid.

Give an example of conjugate acid base.

CONJUGATE ACID-BASE PAIR

CONCEPT –
In any acid-base reaction, there are conjugate acid-base pairs where the acid of one pair is related to the base of another by the transfer of a proton.
EXAMPLE
In the equilibrium reaction $H_2O+NH_3 \rightleftharpoons H_3O^+ + NH_2$, H_2O and H_3O^+ form a conjugate acid-base pair, as do $\mathbf{NH_3}$ and $\mathbf{NH_2}$

Define neutralization curve.

Prepared by- Kiranmayee Bhatra

A neutralization curve is a graphical representation of the pH changes that occur during a titration of an acid with a base or vice versa. The curve typically displays the pH of the solution being titrated as a function of the volume of the titrant (the solution being added). The pH of the solution changes as the titration progresses due to the neutralization reaction between the acid and the base.

Define acidimetry and alkalimetry.

• Acidimetry used to determine the concentration of base substances Using standard acid solution.

• In acidimetry, a known volume of a base is put into a conical flask, the solution is then titrated against a standard solution of acid taken in burette till equivalent point comes.

• Alkalimetry used to determine the concentration of acid substances using standard base.

• In alkalimetry a known volume of an acid is put into a conical flask and then titrated against standard solution of base taken in burette.

• Equivalent point is the paint at which no of moles of analyte (acid) is equal to the number of moles of titrant (base

Write down the difference between aqueous and non-aqueous titration.

Aqueous titration–

These are performed in aqueous solution, (means in water)

Non- aqueous titration

Non -aqueous titration are used when analyte has low water solubility or either weak acid or weak basic in comparison to water.

Define equivalence point.

Definition:

equivalence point is the theoretical point in a titration where the amount of titrant added is stoichiometrically equivalent to the amount of analyte present in the sample. At this point, the reaction is considered complete. The equivalence point does not always coincide exactly with the endpoint, which is the practical indicator used in the titration.

Define titrant.

Titrant

Definition:

The titrant is the solution of known concentration that is added during a titration. It is the reagent that reacts with the analyte (the substance being analysed) to determine its concentration

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MODEL QUESTION AND ANSWER

(10marks)

1) Write a detail note Mohr's, Volhard's and Fajan's method.

Mohr's Method

• The method was given by Sri Karl Friedrich Mohr.

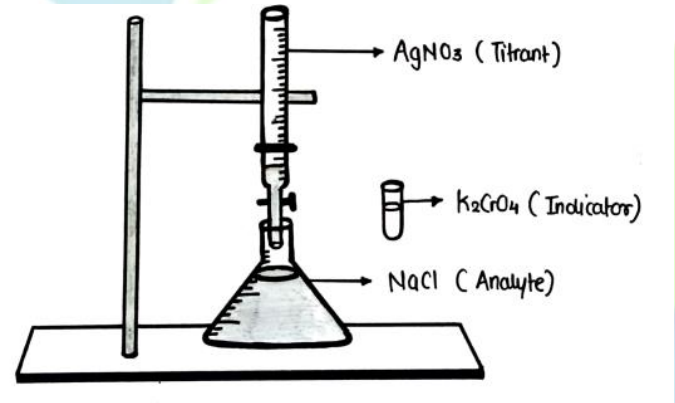
• A precipitation titration in which silver nitrate is used as titrant e chromate ions as indicator is called as Mohr's Method.

• The method is mainly used for the determination of chlorides and bromides (CI and Br).

Titrant: $AgNO_3$

Analyte: KCI, NacI, KBr,

Indicator: $K2Cro_4$ or Na_2 cro_4



Methodology / Procedure

• Let's suppose we are titrating 01 M Nacl using O1M $AgNO_3$ as titrant and Kro_4 as indicator.

• As the titration starts, Silver Nitrate solution is slowly added to Sodium Chloride and a precipitate of Silver Chloride forms.

$Nacl + AgNO_3 \rightarrow AgcI + NaNo_3$

• Now when all the chloride ions get precipitated, then additional silver ion react with chromate ions (indicator) and form a reddish-brown precipitate of silver chromate and this gives our end point.

$$2AgNo_3 + K_2cro_4 \rightarrow Ag_2cro4 + 2KNO_3$$

Conditions For Mohr's Method

• In Mohr's Method titration must be carried out in a neutral medium neither acidic nor basic. Because

1) in basic medium m- $Ag^+ + OH^- \longrightarrow AgOH$

2) in acidic medium - $Cro_4^{2-} + H+ \longrightarrow H_2\ Cro_4$

Note

The Ksp of AgCl = 1.8×10^{-10}

The Ksp of $Ag_2cro4 = 1.2 \times 10^{-12}$

Although Ksp value of Ag2cro4 is lower than ksp value of Agcl and it should be precipitated first, but since the concentration of cl ion in the analyte solution is much higher compare

to Cro_4^{2-}, hence Ag+ react with cl- ions fist f that's' why Agal formed before $Agaro_4$

VOLHARD METHOD

• The method was given by German scientist Jacob Volhard

• This is an indirect method of titration involves back titration.

• Volhard method is mainly used in the determination of Halides [Cl, Bo, I)

• Precipitation titration by Volhard method completed in two steps

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• Now the excess amount of Agro4 which gets remained in

step-1

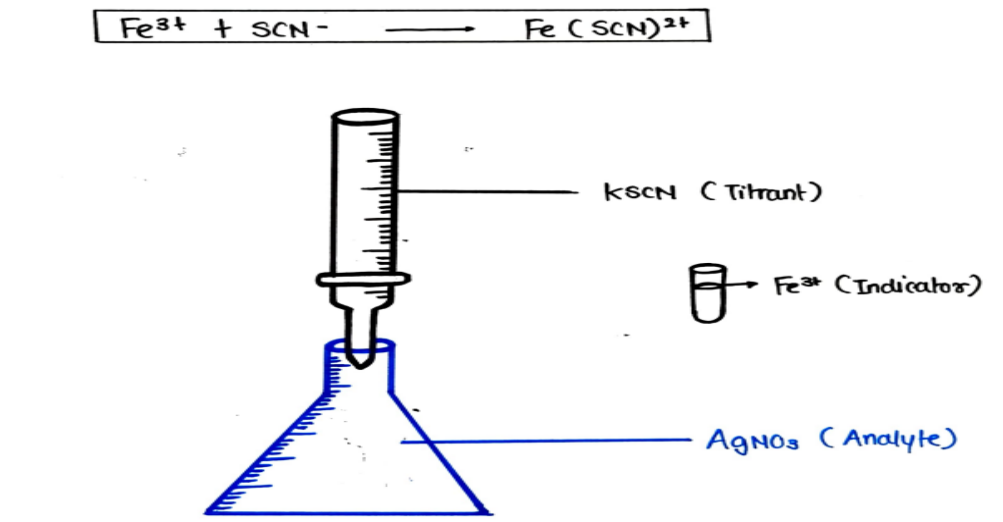
In first step excess amount of Step$AgNO_3$ is used for the titration with NaCl solution.

NaCl + AgNO3 → AgCl + $NaNO_3$ + $AgNO_3$

Step-II

Now the excess amount of $AgNO_3$ which get remains in first step is titrated using kSCN as titrant and $Fe3^{3+}$ as indicator.

AgNO3 + KSCN → AgSCN + KNO3



• Now when all AgNO3 is consumed by KSCN (potassium thiocyanate) then extra thiocyanate ions react with Fe^{3+} and form ferrous thiocyanate ($Fe(SCN)^{2+}$ which is a red complex and this gives our end point.

$Fe^{3+} + SCN^{-} \rightarrow Fe(SCN)^{2+}$

Conditions for Volhard's Method

The titration must be performed in acidic medium, so the complex formed $Fe(SCN)^{2+}$ will be stable.

MODIFIED VOLHARDS' Method

• in modified Volhard's

method we add some wetting agents like chloroform, nitrobenzene etc.

• Since. AgCl is also present in the analyte and it may solubilize which affect our end point and addition of these wetting agents prevents the solubilization of Agcl.

FAJAN METHOD

• The method was given by sir Kazimierz Fajan.

• The method is based upon the theory adsorption indicators.

• The method is used for the determination of halides (CI, Bo, I) using AgNo3 as titrant and Fluorescein as indicator.

• At the end point, adsorption indicators get adsorb on the surface of Agcl and changes the colour of precipitate.

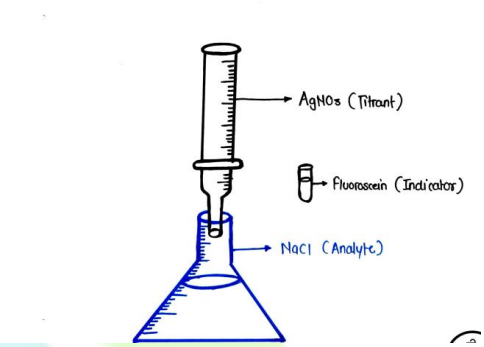
Titrant- : $AgNO_3$

Analyte: Nacl

Indicator: Fluorescein

$AgNO3 + NaCl \rightarrow AgCl + NaNO_3$

\



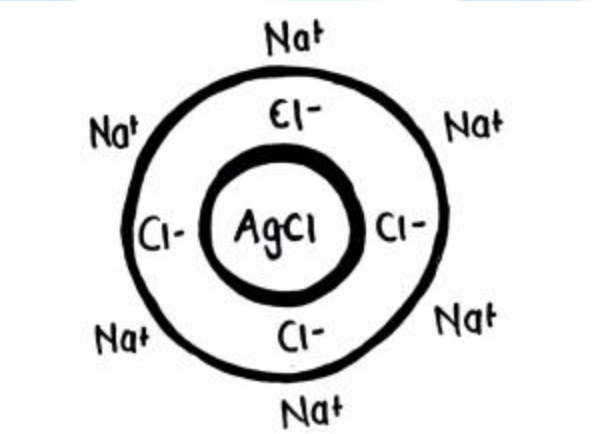
Procedure

When all the NaCl solution in the analyte is consumed by AgNo3 and converted into Agcl, then fluorescein indicator gets adsorption the surface of Agcl e changes the colors to pink.

Before Endpoint

AgNO3 reacts with NaCl and converted into AgCl.

$AgNO3 + NaCl \rightarrow AgCl + NaNO_3$



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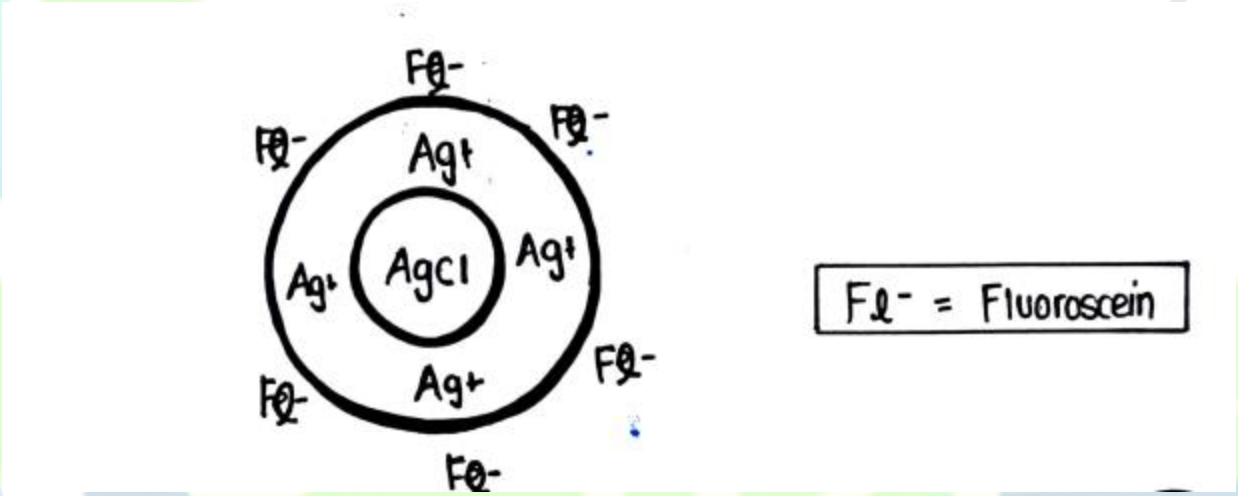
At Point

Fluorescein indicator adsorbs on the surface of AgCl and change the colour.

Ag^{+} + Fl^{-} → AgFl



2) Describe about masking and de-masking agent.

Masking& Demasking Agents

• Masking and demasking agents are generally used when analyte solution contains two or more than two metal ions.

• Ler consider we have a solution containing three different metal ions A, B and C , now it we directly titrate the analyte, then we will get the concentration of al there metals A, CB,

• But in the case if we have to determine the concentration of A,8 and C individually then we have to use Masking and Demasking Agents.

Masking Agents

• Masking agents are the reagents that forms a complex with some components of the analyte and protect them from reaction with EDTA

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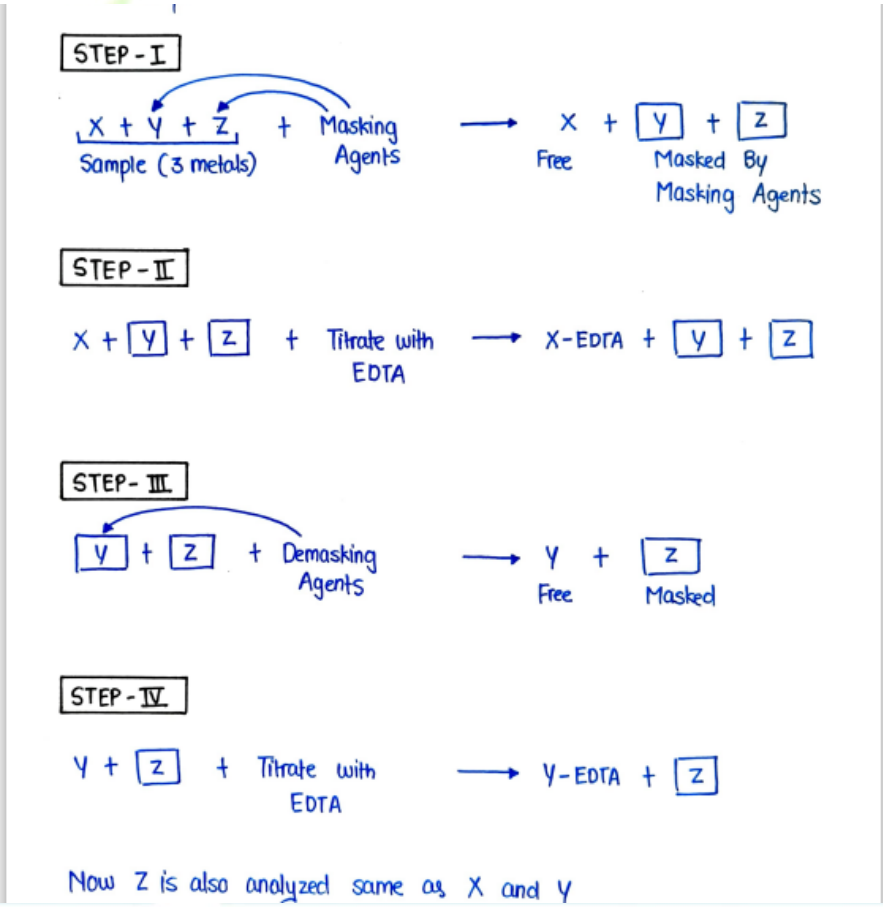
• Masking agents air mainly used when the analyte solution contains two or more metal ions

Demasking Agents

• Demasking agents are the reagents that releases metal ions from masking agents.

• They break the complex formed between metal ion masking agent

Mechanism of masking and de-masking agent



Lets consider we have an analyte solution containing three metal ions X. y and I and we have ot find their concentration

Masking Agent: Ethylenediaminetetraacetic Acid (EDTA)

Demasking Agent: Sulfuric Acid (H2SO4)

Reaction Example:

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1. **Masking Reaction (Masking of Calcium Ions):**

- **Masking Agent (EDTA) reacts with Calcium Ions (Ca^2+):**

$Ca^{2+} + EDTA \rightarrow [Ca(EDTA)]^{2-}$

- In this reaction, EDTA forms a stable complex with calcium ions, preventing them from interfering with the analysis of the target pharmaceutical compound.

2. **Demasking Reaction (Demasking of Calcium Ions):**
   - **Demasking Agent (Sulfuric Acid) reacts with [Ca(EDTA)]**

$[Ca(EDTA)]^{2-} + H_2SO_4 \rightarrow CaSO_4\downarrow + EDTA$

   - Sulfuric acid breaks the complex between EDTA and calcium ions, releasing calcium ions in the form of calcium sulphate ($CaSO_4$) and regenerating the free EDTA.

By incorporating these masking and demasking steps, the pharmaceutical analyst can effectively control interference from calcium ions and ensure accurate quantification of the target analyte.

This example illustrates how masking and demasking play crucial roles in pharmaceutical analysis, allowing for the reliable determination of active pharmaceutical ingredients in the presence of potential interferents.

3) Describe the principle and steps involved in gravimetric analysis.

GRAVIMETRIC ANALYSIS

• The word gravimetry refers to mass.

• Gravimetric analysis is method of analysis in which we determine the quantity of analyte by measurement of mass.

• In gravimetric analysis analyte is converted into precipitate.

Methods of gravimetric

- Precipitation Gravimetry
- Volatilization Gravimetry
- Electrogravimetry

Advantages of Gravimetric Analysis

- Gravimetric analysis is more accurate and more precise compared to volumetric analysis.
- It is an absolute method which involves direct measurement.
- Less chances of mistake.
- Generally, not affected by temperature.

Disadvantages of Gravimetric Analysis

- It is very time consuming.
- It should be performed very carefully.
- It requires very clean glassware and very accurate weighing.

STEPS NIVOLVEDINGRAVIMETRICAMALVSIS

The estimation of gravimetric analysis consists a number of steps to get accurate results, it includes

- Sampling
- Preparation of solution
- Precipitation
- Digestion / Ostwald Ripening
- Filtration
- Washing
- Drying or Ignition
- Weighing
- Calculations

[Sampling

• The sample which very small. is weighed for gravimetric analysis

• The sample should be homogenous.

• The sample must be in powder form.

• It should not be very expensive.

Preparation of Solution

• to dissolve the sample, take a clean beaker and transferred the weighed sample completely into beaker.

• Ad sufficient water to get a cleat sample.

• temperature, pH, pressure should be maintained at normal

• Iff necessary, then heat the solution.

Precipitation

• in this process, we generally ad a precipitating agent to the sample solution.

- The ideal precipitating agent must react with analyte to form precipitate.
- Precipitation generally caried out in a resistance glass beaker.
- The completeness is checked by adding a few drops of precipitating agent by the side of beaker wall.
- it is very necessary to check the completeness of precipitation.

Digestion

• Digestion process is also known as Ostwald Ripening.

• During digestion, the precipitate is left hat f o r 30 min- + have

• Digestion involves dissolution of small particles.

• Digestion process is very helpful in the case of colloidal particles.

• Digestion, sometimes increases the problem of post -precipitation.

Filtration

• in this step, precipitate is separated from the analyte solution.

• Various types of filer media used in this process.

• The choice of filter media is depended upon the nature of precipitate.

• The different filer media used in gravimetric analysis are O

- Filter Paper
- Filer Pulp

- filler Mats etc.

Washing

• After filtering the precipitate, the impurities at the surface of the precipitate can be removed by washing the precipitate.

• One should be very careful not to use too much water during washing, because part of the precipitate may lost.

• water is not suitable for many precipitates as washing solution.

• in such cases dilute nitric acid, ammonium nitrate etc. can be used as washing solution.

Drying ignition

• The mani purpose of drying or ignition is to convert the precipitate in a constant composition.

• It is heated to remove the water

• Drying team is used when temperature is below 250 c

• Ignition term is used when temperature is above 250°6 and below 1200° C.

• Drying or Ignition is depended upon het nature of precipitate.

Weighing and Calculation

- After drying the precipitate is then cooled at room temperature and weighed accurately on the analytical balance.
- The calculations are generally made in teams of percentage.
- Calculations of analyte content requires knowledge of
- -Chemistry
- Stoichiometry
- Composition of precipitate

(5marks)

1) Define the principle method and application of diazotization titration.

DIAZOTISATION TITRATION

• Diazotisation titration is a method used for determination of primary aromatic amine compounds.

• The diazotisation titration is nothing but simply the determination of primary aromatic amine to a diazonium compound.

• The process was first discovered by Peter Griessin in 1853.
• The method is mainly used for determination of dyes.

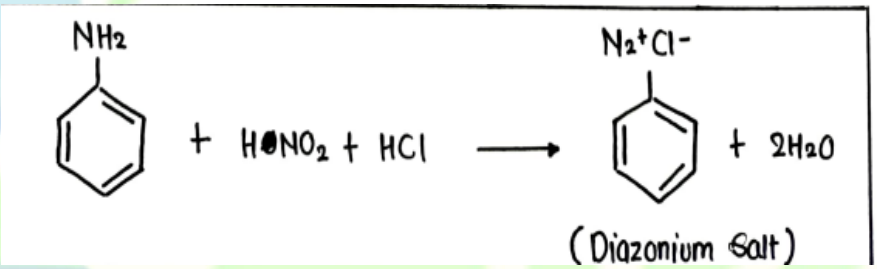
Principle of Diazotisation Titration

• Primary aromatic amines in the presence of Hal reacts with Sodium Nitrite (NaNO3) and form Diazonium salt
• The reaction completed in two steps

STEP - I

$NaNO_2 + HCl \rightarrow HNO_2 + NaCl$
+ Naci
Sodium Nitrate (NaNO3) react with HCI and form Nitrous Acid (HNO_2) and Sodium Chloride (Nacl)

STEP – II



The obtained nitrous acid reacts with Diazonium Salt.

End Point Determination in Diazotisation Titration

• In diazotisation titration we use starch Iodine Paper as indicator.
• At the end point of the titration, starch iodine paper changes the colour into blue.

Starch Iodine Paper

• Starch iodine paper is prepared by dipping the filter paper into Potassium iodide (KI) and starch mucilage solution.
• Following reaction occur during end point determination

$$KI + HCl \rightarrow HI + KCl$$

$$2HI + 2HNO_2 \rightarrow I_2 + 2NO + 2H_2O$$

$$I_2 + \text{starch} \rightarrow I_2 \text{ starch complex blue}$$

Condition For Diazotisation Titration

• Maintenance of temperature is the main condition for diazotisation titration
•

• The diazonium salt is not stable at high temperature, so the temperature should be maintained at 0-5°6.

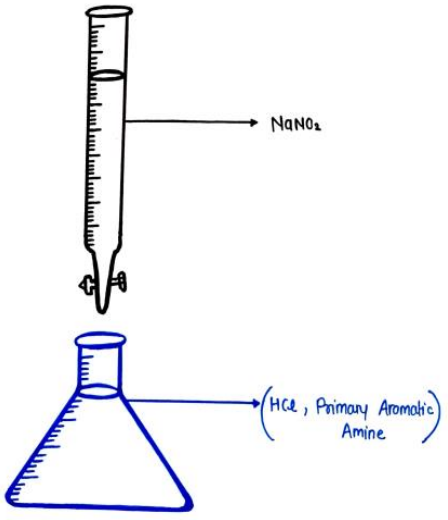
Disadvantages of Diazotisation Titration

• Applicable for very les variety of samples.
• Relatively slow compared to other methods
• Required to maintain temperature conditions.
• End point detection is very difficult

Methodology

• The general procedure is followed by weighing the sample and transferring it into standard flask.

• After that concentrated hydrochloric acid and potassium bromide are added and rest of the volume is filled with distilled water.
• The resulting solution is known as standard solution.
• The appropriate volume of the standard solution is pipettes out and temperature is maintained at 0-5°6
• Now finally the solution is titrated with sodium nitrite solution until starch iodide paper tons into blue colour.



Applications of Diazotisation Titration

• Used in the determination of sulphonamides
• Used in the determination of chlorpheniramine
• Used in the determination of procaine
• Used in the determination of dopamine
• Used In the determination of ephedrine etc

.

2) Describe briefly about Pm indicators.

Metal Ion Indicators

• Indicators that are used in complexometric titration are known as Metal Ton Indicators.
• It is also known as PM Indicators

Mechanism of Action PM Indicators

• Like all other indicators, MP indicators also shows colour change at the endpoint, but its working is somehow different: -
• First, we add indicator in the analyte (metal ions) solution, now indicator formed a complex with analyte and colour of this complex is wine red, hence we can say before end point the colour of the analyte is wine red.
• A t Endpoint: At the Endpoint, the titrant (ligands) breaks the bond between indicator and analyte and titrant self-forms a complex with analyte which is called Metal Ion Complex or Ligand- metal ion complex.

• How since indicator gets free, and the free form of indicator have blue colour, hence we can say at the endpoint, indicator Shows blue colour

• Some commonly used indicators in complexometric titrations are: Eriochrome black T

, Calmagite, Murexide Catechol Violet etc.

3) Write down the estimation of Barium sulphate.

Estimation OF Barium Sulphate

•Chemical Formula
Molecular Weight- 233.4 g/mol

Properties

• it is a fine, heavy, white colour powder
• It is odourless
• It is tasteless

• It is insoluble in water

Principle

When dilute Sulphuric acid (H2SO4) is added to the dilute solution of barium chloride (Bacl2) a white precipitate of barium sulphate (BaSO4) is formed.

$$Bacl2 + H2SO4 \rightarrow Baso4 + 2Hcl$$

Procedure

• Pipette out 25 ml of given solution of barium chloride in 500 ml beaker.
• Now add 0.5 ml concentrated H2so4 and 100 ml distilled water.

• If required then heat the solution.
• Now to this solution add dilute sulphuric acid (H2S04) dropwise until the precipitate of Baso4 (barium sulphate) is not formed
• Allow the precipitate to settle down.

• Now filter the precipitate.
• Wash the precipitate 3-4 times with hot water.
• Now dry out the precipitate
• If water is not removed then repeat the process of drying /Ignition.
• Now cool the precipitate, measure the weight and calculate the amount of barium.

4) Describe briefly about complexometric titration.

COMPLEXOMETRIC TITRATION

• Complexometric titration is a type of titration which is based on the complex formation between the analyte and titrant.

• It is also known as Chelatometric Titration.

• Complexometric Titration are mainly used for the determination of metal ions in a solution.

• End point of the titration is determined by change in colour of the solution due to complexometric reaction.

• Since EDTA (Ethylene diamine tetra acetic acid is mainly used as titrant in complexometric titration, hence it is also called EDTA Titration.

in Complexometric Titration We can simply say:

Analyte: Metal Ions

Titrant: Ligands or Complexing Agents (Mainly EDTA)

Indicator: Metal Ion Indicators

Types of Ligands in Complexometric Titration

Ligands are classified on the basis of number of donor groups:

- Unidentate Ligands / Monodentate Ligands
- Bidentate Ligands
- Multidentate / Polydentate Ligands

Monodentate Ligands

• It is also known as unidentate Ligands.

• When the ligands have only one donor atom, it is called monodentate.

examples: NH_3, H20 etc.

Bidentate Ligands

• When the ligands have two donor atoms, it is called Bidentate ligands

• example: Ethylene Diamine (H_2N: - CH2 -CH2 – NH_2)

Multidentate Ligands

• It is also known as Polydentate ligands.

• When the ligands have more than 2 donor atoms, it is called as Multidentate ligands.

• example EDTA (Ethylene Diamine Tetra acetic Acid)

• Möno dentate ligands are also known as Chelates

:

Classification complexometric Titration

• Direct Titration

• Back Titration / Indirect Titration

• Replacement Titration

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• Alkalimetric Titration

Direct Titration

• It is the simplest method of complexometric titration.

• in this EDTA is mainly used as titrant.

• In this end point is simply determined by colour change due to indicator.

• It is similar to acid- base titration.

• It is not suitable for slow complexometric titrations.

Direct Titration of Hydrochloric Acid with Sodium Hydroxide:

Reaction: $HCl + NaOH \rightarrow NaCl + H_2O$

Explanation:

1. **Setup:**
   - A known volume of hydrochloric acid (HCl) is taken in a flask and titrated with a solution of sodium hydroxide ($NaOH$) of known concentration.
   - The goal is to determine the concentration of the hydrochloric acid solution.
2. **Titration Process:**
   - Sodium hydroxide ($NaOH$) is slowly added to the hydrochloric acid (HCl) solution using a burette.
   - The reaction between hydrochloric acid and sodium hydroxide is a neutralization reaction.

Back Titration

• It is also known as Indirect titration.

• in this titration is performed by taking excess amount of EDTA

•in this first we add excess amount of EDTA in the analyte, then remaining amount of EDTA is again titrated by solution of second metal ion.

Back Titration Example: Determination of Calcium Carbonate in an Antacid Tablet

Reaction Steps

1. **Reaction with Excess Hydrochloric Acid (HCl):**
   $CaCO_3 + 2HCl \rightarrow CaCl_2 + CO_2 + H_2O$
   - Calcium carbonate ($CaCO_3$) in the antacid tablet reacts with excess hydrochloric acid ($2HCl$).
   - Carbon dioxide (CO_2) is evolved during the reaction.
2. **Excess Acid Back Titration with Sodium Hydroxide (NaOH):**
   $2NaOH + CO_2 \rightarrow Na_2CO_3 + H_2O$
   -
   - The remaining excess hydrochloric acid is neutralized by adding sodium hydroxide ($2NaOH$).
   - The endpoint is reached when all excess acid is neutralized.

Replacement Titration

• This method is used when direct &back titration don't' give sharp end paint.

• In this titration, metal present in the analyte displaces another metal from metal - EDTA complex.

Replacement Titration Example: Determination of Sodium Hypochlorite (NaClO) Concentration

Reaction Steps:

1. **Oxidation of Iodide Ions (I^-) by Sodium Hypochlorite (NaClO):**
   $2I^- + 2H^+ + NaClO \rightarrow I_2 + NaCl + H_2O$
   - Sodium hypochlorite oxidizes iodide ions to iodine (I_2).
   - The iodine formed is not stable in solution and reacts with starch to give a blue colour.
2. **Replacement Titration with Sodium Thiosulfate ($Na_2S_2O_3$):**
   $Na_2S_2O_3 \rightarrow 2NaI + Na_2S_4O_6$
   - Sodium thiosulfate is added to the solution, and it reacts with the iodine produced in the first step.
   - The endpoint is reached when all the iodine is reduced by sodium thiosulfate.

Alkalimetric Titration

• It is used for the determination of anions which generally do not react with EDTA chelate.

• in this method, the free its ions get liberated during titration.

• The free ions are then titrated with standard solution of alkali Using suitable indicator.

Alkalimetric Titration Example: Titration of Hydrochloric Acid (HCl) with Sodium Hydroxide (NaOH)

Reaction Steps:

1. **Titration Setup:**
   - A known volume of hydrochloric acid solution is taken in a flask.
   - Sodium hydroxide solution of known concentration is used as the titrant and is slowly added to the acid solution.
2. **Neutralization Reaction:**

$$HCl + NaOH \rightarrow NaCl + H_2O$$

- Hydrochloric acid (HCl) reacts with sodium hydroxide ($NaOH$) in a 1:1 ratio.
- The reaction results in the formation of sodium chloride ($NaCl$) and water (H_2O).
- This is a neutralization reaction where the acidic solution is neutralized by the alkaline solution.

(2 marks)

1) Define complexometric titration.

• Complexometric titration is a type of titration which is based on the complex formation between the analyte and titrant.

• It is also known as Chelatometric Titration.

• Complexometric Titration are mainly used for the determination of metal ions in a solution.

• End point of the titration is determined by change in colour of the solution due to complexometric reaction.

• Since EDTA (Ethylene diamine tetra acetic acid is mainly used as titrant in complexometric titration, hence it is also called EDTA Titration

2) Define masking and de- masking agent.

Masking Agents

• Masking agents are the reagents that forms a complex with some components of the analyte and protect them from reaction with EDTA

• Masking agents air mainly used when the analyte solution contains two or more metal ions

Demasking Agents

• Demasking agents are the reagents that releases metal ions from masking agents.

3)Define precipitation titration.

• The word precipitation refers to formation of solid mas in a liquid.

• The solid formed is termed as Precipitate.

• Precipitation titration is a type of titration which involves the formation of precipitate during titration.

• Since, precipitation titration is mainly used $AgNO_3$ (Silver Nitrate) as titrant, hence it is also called "Argentometric Titration:

3) Write down the difference between co- precipitation and post-precipitation.

Difference between co- precipitation and post-precipitation

	Co- precipitation	**Post- precipitation**
Definition	Co-precipitation involves the unintentional inclusion of impurities or undesired components during the precipitation of a target analyte.	Post-precipitation refers to the precipitation of a substance after the primary precipitation of the target analyte.
Degree of contamination	High	Low
Times of precipitation	During the precipitation	After the precipitation

4) What are the type of co- precipitation.

Co- precipitate

is a process in which during the precipitation, impurities also get precipitated along with our main precipitate

• The problem of co-precipitation usually occurs when more than 2-3 compounds are mixed ni our analyte solution.
• The chances of co-precipitation are generally higher during gravimetric analysis

Types/Mechanism of Co- Precipitation

- Inclusion
- Occlusion
- Surface Adsorption
- Mechanical Entrapment

5) Define diazotization titration.

• Diazotisation titration is a method used for determination of primary aromatic amine compounds.
• The diazotisation titration is nothing but simply the determination of primary aromatic amine to a diazonium compound.
• The process was first discovered by Peter Griessin in 1853.
• The method is mainly used for determination of dyes.

6) What do you mean by gravimetric analysis.

• The word gravimetry refers to mass.
• Gravimetric analysis is method of analysis in which we determine the quantity of analyte by measurement of mass.
• In gravimetric analysis analyte is converted into precipitate.

JRG College of Pharmacy

MODEL QUESTION ANSWER OF REDOX TITRATION

UNIT- IV

(10 MARKS)

- **Explain the types of redox titration.**

Redox titration is a type of titration that involves a redox (reduction-oxidation) reaction between the analyte (the substance whose concentration is being determined) and the titrant (the solution of known concentration). In redox titrations, the transfer of electrons between the reactants is what drives the reaction to completion.

- **Cerimetry**:
  Cerimetry is a type of titration where cerium^{4} sulphate is used as a titrant. It's particularly useful for the determination of reducing agents. Here's an example:
  Example: Determination of iron in iron supplements
  In this titration, a known volume of iron solution is titrated with cerium sulphate solution until the endpoint is reached. The endpoint is detected by the colour change from yellow to colourless or faint pink. The reaction involved is:
  $Fe2++Ce4+\rightarrow Fe3++Ce3+Fe^{2+}+Ce^{4+}\rightarrow Fe^{3+}+Ce^{3+}$
  By knowing the volume and concentration of the cerium(IV) sulphate solution used, the concentration of iron in the sample can be calculated.
- **Iodimetry Titration**:
  Iodimetry titration involves the use of iodine as a titrant to determine the concentration of oxidizing agents. Here's an example:
  Example: Determination of vitamin C (ascorbic acid) in a pharmaceutical formulation
  In this titration, a known volume of ascorbic acid solution is titrated with iodine solution until the colour changes from colourless to faint yellow. The reaction involved is:
  $\text{Ascorbic acid}+I2\rightarrow\text{Dehydroascorbic acid}+2HI\text{Ascorbic acid}+I_2 \rightarrow\text{Dehydroascorbic acid}+2HI$
  The amount of iodine solution used is then used to calculate the concentration of ascorbic acid in the sample.
- **Iodometric Titration**:
  Iodometric titration involves the use of iodine generated by the reaction of

an oxidizing agent with potassium iodide. Here's an example:
Example: Determination of copper in brass

In this titration, a known volume of copper solution is reacted with excess potassium iodide to form a complex. The excess iodine liberated is then titrated with a sodium thiosulfate solution until the blue colour disappears. The reaction involved is:

$2Cu2++4I-\rightarrow 2CuI+I22Cu^{2+}+4I^{-}\rightarrow 2CuI+I_2$

The amount of thiosulfate solution used is then used to calculate the concentration of copper in the sample.

- **Bromometry**:
  Bromometry involves the use of bromine as a titrant. It's commonly used in the determination of reducing agents. Here's an example:
  Example: Determination of sulphite ions in wine
  In this titration, a known volume of wine is titrated with bromine water until the endpoint is reached. The endpoint is detected by the decolorization of the bromine colour. The reaction involved is:
  $SO32-+Br2\rightarrow SO42-+Br-SO_3^{2-}+Br_2\rightarrow SO_4^{2-}+Br^{-}$
  The concentration of sulphite ions in the wine can then be calculated based on the volume and concentration of the bromine solution used.
- **Dichrometry Titration**:
  Dichrometry titration involves the use of dichromate ions as a titrant. It's often used in the determination of reducing agents. Here's an example:
  Example: Determination of ferrous ions in iron supplements
  In this titration, a known volume of ferrous ion solution is titrated with dichromate solution until the endpoint is reached. The endpoint is detected by the change in colour from orange to green. The reaction involved is:
  $Fe2++Cr2O72-+14H+\rightarrow Fe3++2Cr3++7H2OFe^{2+}+Cr_2O_7^{2-}+14H^{+}\rightarrow Fe^{3+}+2Cr^{3+}+7H_2O$
  By knowing the volume and concentration of the dichromate solution used, the concentration of ferrous ions in the sample can be calculated.

- **Describe about polarography**

Polarography is a sophisticated electroanalytical technique used for the

qualitative and quantitative analysis of various substances in solution. Developed by Jaroslav Heyrovsky in the early 20th century, polarography relies on the measurement of the current flowing through an electrochemical cell as a function of an applied voltage.

Here's a detailed description of polarography:

- **Principle**: Polarography is based on the principle of polarographic reduction. In this technique, a dropping mercury electrode (DME) is used as the working electrode, immersed in the solution containing the analyte. A potential is applied to the electrode, causing it to oscillate between two predetermined values. As the potential changes, reduction or oxidation reactions occur at the electrode surface, resulting in changes in the current flowing through the cell. By measuring this current as a function of the applied potential, information about the analyte's concentration and nature can be obtained.
- **Instrumentation**: Polarographic instruments typically consist of a potentiator, which controls the potential applied to the working electrode, and a reference electrode (e.g., a saturated calomel electrode) to provide a stable reference potential. The working electrode is a dropping mercury electrode (DME) or a rotating disk electrode (RDE), depending on the specific application. The instrument also includes a stirrer to ensure uniform mixing of the solution.
- **Operation**: During a polarographic experiment, the potential applied to the working electrode is varied linearly or in steps. As the potential changes, reduction or oxidation of the analyte occurs at the electrode surface. The resulting current is measured using an ammeter connected in series with the cell. The current-potential curve obtained is called a polarogram.
- **Applications**: Polarography finds applications in various fields, including pharmaceuticals, environmental analysis, and metallurgy. It is particularly useful for the determination of metal ions, organic compounds, and electroinactive species. Common applications include the analysis of heavy metals in environmental samples, the determination of trace metals in biological fluids, and the quantification of organic compounds in pharmaceutical formulations.
- **Advantages**:
- High sensitivity: Polarography can detect trace amounts of analyte in solution,

making it suitable for the analysis of dilute samples.

- Wide applicability: Polarography can be applied to a wide range of substances, including inorganic ions, organic compounds, and gases.
- Rapid analysis: Polarographic measurements are often quick and straightforward, allowing for high-throughput analysis of multiple samples.
  - **Limitations**:
- Mercury contamination: The use of mercury electrodes in polarography can lead to environmental concerns due to the toxicity of mercury.
- Limited selectivity: Polarography may suffer from interference from other substances present in the sample, particularly in complex matrices.

- Skill requirement: Interpretation of polarograms and optimization of experimental conditions require expertise and experience.

(5 MARKS)

- **Explain the concept of redox titration.**

Redox titration is a fundamental analytical technique used to determine the concentration of a substance (the analyte) in a sample through a redox reaction. In redox titrations, the analyte undergoes a redox reaction with a titrant of known concentration. The endpoint of the titration is determined by monitoring a change in the oxidation state of the analyte, which is often detected using an indicator, a potentiometric method, or through instrumental analysis.

The concept of redox titration revolves around the principles of oxidation and reduction reactions. Oxidation involves the loss of electrons, while reduction involves the gain of electrons. During a redox titration, the analyte is either oxidized or reduced by the titrant, leading to a measurable change in the oxidation state of the analyte.

The key components of a redox titration include the analyte, the titrant, and an indicator or detection method. The titrant is chosen based on its ability to undergo a redox reaction with the analyte. Common titrants include potassium permanganate (KMnO4), potassium dichromate (K2Cr2O7), iodine (I2), and cerium (IV) sulphate (Ce (SO4)2), among others.

The endpoint of the titration is the point at which the stoichiometric amount of titrant has reacted with the analyte. This is typically indicated by a colour change in the solution (if

using an indicator), a change in potential (if using potentiometric methods), or through instrumental techniques such as spectrophotometry.

Redox titrations are widely used in various industries, including pharmaceuticals, environmental analysis, and food chemistry. In pharmaceutical analysis, redox titrations are employed to determine the concentration of active pharmaceutical ingredients, impurities, and degradation products in drug formulations.

Overall, redox titration is a versatile and powerful analytical technique that provides quantitative information about the concentration of substances in a sample based on their redox behaviour. Its simplicity, accuracy, and wide applicability make it an indispensable tool in analytical chemistry.

- **Explain dichrometry.**

Dichrometry is a type of redox titration that involves the use of dichromate ions ($Cr2O72-Cr_2O_{72-}$) as the titrant. It is commonly used to determine the concentration of reducing agents in a sample. Dichromate ions are powerful oxidizing agents and can be reduced to chromium(III) ions ($Cr3+Cr_{3+}$) in acidic medium. The reduction of dichromate ions by the analyte is a key step in dichrometry.

Here's a detailed explanation of dichrometry along with an example reaction:

Principle: In dichrometry, the analyte containing the reducing agent is titrated with a solution of dichromate ions. The reducing agent in the analyte undergoes oxidation, while the dichromate ions are reduced to chromium (III) ions. The endpoint of the titration is reached when all the reducing agent in the sample has been oxidized by the dichromate ions.

Example Reaction: Determination of Ferrous Iron ($Fe2+Fe_{2+}$) in a Sample

Reaction: $6Fe2++Cr2O72-+14H+→6Fe3++2Cr3++7H2O6Fe_{2+}+Cr_2O_{72-}$

$+14H^{+} \rightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O$

Explanation:

- **Preparation of Sample**: The sample containing ferrous ions ($Fe2+Fe^{2+}$) is prepared by dissolving it in an appropriate solvent.

- **Titration Setup**: A measured volume of the sample solution is placed in a flask. Dilute sulfuric acid ($H2SO4H_2SO_4$) is added to provide an acidic medium, which facilitates the reaction between the analyte and the titrant.

- **Titration Process**: A solution of potassium dichromate ($K2Cr207K_2Cr_2O_7$) of known concentration is slowly added to the sample solution using a burette. The dichromate ions ($Cr2072-Cr_2O_{72-}$) in the titrant oxidize the ferrous ions ($Fe2+Fe^{2+}$) in the sample to ferric ions ($Fe3+Fe^{3+}$). The reaction is highly exothermic and produces a green colour due to the formation of chromium(III) ions.

- **Endpoint Detection**: The endpoint of the titration is detected using an indicator or a suitable method. In dichrometry, the endpoint is typically detected by a change in colour from orange to green, indicating the complete oxidation of ferrous ions to ferric ions.

- **Calculations**: The volume of the titrant consumed at the endpoint is used to calculate the concentration of the reducing agent (ferrous ions) in the sample solution. This is done using the stoichiometry of the reaction and the known concentration of the titrant.

Applications: Dichrometry is commonly used in various industries, including pharmaceuticals, environmental analysis, and metallurgy, for the determination of reducing agents such as ferrous ions, sugars, and organic compounds.

- **Write a note on iodometry.**

Iodometry is a volumetric titration method based on the oxidation and reduction reactions involving iodine and iodide ions. It is commonly used in analytical chemistry for the quantitative determination of oxidizing agents, particularly those that cannot be directly titrated with standard solutions of reducing agents.

Principle: Iodometry relies on the reaction between iodine (I2) and iodide ions (I^-) in an acidic medium to produce triiodide ions (I_3^-), according to the equation:

$$I^- + I2 \rightleftharpoons I3^- \quad I^- + I_2 \rightleftharpoons I_3^-$$

The concentration of iodine or iodide ions in the solution can be determined by titrating them with a standard solution of a reducing agent, such as thiosulfate (Na2S2O3), which converts iodine or iodide ions back to iodide ions:

$$I2 + 2S2O3^{2-} \rightarrow 2I^- + S4O6^{2-} \quad I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-}$$

The endpoint of the titration is detected using an indicator that undergoes a color change in the presence of excess iodine, such as starch, which forms a blue complex with iodine.

Applications: Iodometry finds numerous applications in analytical chemistry, including:

- **Determination of Oxidizing Agents:** Iodometry is commonly used to determine the concentration of oxidizing agents such as chlorine, bromine, and permanganate ions in various samples.

- **Water and Wastewater Analysis:** Iodometric titration is employed in environmental analysis for the determination of chlorine content in water and wastewater treatment processes.

- **Pharmaceutical Analysis:** Iodometry is utilized in pharmaceutical analysis for the quantification of pharmaceutical substances containing oxidizing agents or for the assay of certain drugs.

- **Food and Beverage Industry:** Iodometry is applied in the food and beverage industry for the determination of certain additives, preservatives, and antioxidants in food products.

Advantages:

- Iodometry offers high sensitivity and precision in the determination of oxidizing agents.
- It can be applied to a wide range of samples and analytes.
- The method is relatively simple and straightforward to perform with standard laboratory equipment.

Limitations:

- Iodometry may be susceptible to interferences from other substances present in the sample.
- The reaction kinetics may be influenced by factors such as pH, temperature, and the presence of catalysts.
- Care must be taken to ensure accurate endpoint detection, as the color change with starch indicator can sometimes be subtle.

In conclusion, iodometry is a valuable analytical technique with widespread applications in quantitative chemical analysis, particularly for the determination of oxidizing agents in various fields of science and industry.

- **Explain indicators used in redox titration.**

Indicators used in redox titrations serve the same fundamental purpose as indicators in

acid-base titrations: they signal the endpoint of the titration. However, in redox titrations, the endpoint is determined by a change in the oxidation state of the analyte or titrant rather than a change in pH. Here are some common types of indicators used in redox titrations:

- **Potassium Permanganate ($KMnO_4$):** Potassium permanganate is a widely used indicator in redox titrations, particularly for the determination of reducing agents. It is a strong oxidizing agent and undergoes a colour change from purple to colourless (or light pink) upon reaction with reducing agents. The endpoint is typically indicated by the disappearance of the pink colour of the permanganate solution.

- **Starch-Iodine Complex:** Starch is often used as an indicator in iodometric titrations, where iodine is the titrant. Starch forms a deep blue complex with iodine, but the complex is disrupted in the presence of excess reducing agent, leading to a colour change from blue to colourless. The disappearance of the blue colour indicates the endpoint of the titration.

- **Dichromate Ion ($Cr_2O_7^{2-}$):** Dichromate ion is commonly used as an indicator in titrations involving ferrous ions (Fe^{2+}) or other reducing agents. In acidic solutions, dichromate ions are reduced to chromium (III) ions (Cr^{3+}) by the analyte. The endpoint is signalled by the colour change of the solution from orange (dichromate) to green (chromium (III) ion).

- **Diphenylamine Sulfonic Acid:** This indicator is used in titrations involving sulphate ions (SO_3^{2-}) as the analyte. Diphenylamine sulfonic acid forms a blue complex with sulphate ions, and the appearance of a blue colour indicates the endpoint of the titration.

- **Methylene Blue:** Methylene blue is used as an indicator in redox titrations involving oxygen as the oxidizing agent. It is colourless in its reduced form but turns blue upon oxidation. The appearance of a blue colour indicates the endpoint when all the reducing agent has been consumed.

- **Ferroin:** Ferroin is a complex of iron (II) ions with 1,10-phenanthroline. It is often used in redox titrations involving iron (II) and iron (III) ions. The colour of ferroin changes from pale violet (in the oxidized form) to reddish-orange (in the

reduced form) upon oxidation by iron (III) ions, indicating the endpoint.

These indicators are selected based on their sensitivity to specific redox reactions and their ability to produce a clear and distinct color change at the endpoint of the titration. Proper selection and use of indicators are crucial for accurate and reliable results in redox titrations.

- **Explain redox potential.**

- **Definition**: Redox potential, short for reduction-oxidation potential, is a measure of the tendency of a chemical species to undergo reduction or oxidation in a chemical reaction. It quantifies the affinity of a substance for electrons, indicating its readiness to gain or lose electrons.

- **Electron Transfer**: Redox reactions involve the transfer of electrons from one chemical species (the reducing agent) to another (the oxidizing agent). The reducing agent donates electrons and is oxidized, while the oxidizing agent accepts electrons and is reduced.

- **Measurement**: Redox potential is measured in volts (V) or millivolts (mV). It is typically determined using a reference electrode, such as the standard hydrogen electrode (SHE) or a saturated calomel electrode (SCE), in combination with the substance of interest.

- **Standard Redox Potential (E°)**: The standard redox potential, denoted as E°, is the redox potential of a half-reaction under standard conditions (1 M concentration, 1 atm pressure, and a specified temperature, often 25°C or 298 K). It serves as a reference point for comparing the redox potentials of different reactions.

- **Nernst Equation**: The Nernst equation relates the redox potential (E) of a half-reaction to the concentrations of the reactants and products involved. It is expressed as:
$E=E\circ-RTnF\ln\ ([oxidized][reduced])E=E\circ-nFRT\ln([reduced][oxidized])$

Where:

- EE is the redox potential.
- $E∘E_∘$ is the standard redox potential.
- RR is the gas constant (8.314 J/(mol·K)).
- TT is the temperature in Kelvin.
- nn is the number of moles of electrons transferred.
- FF is the Faraday constant (96,485 C/mol).
- $[oxidized][oxidized]$ and $[reduced][reduced]$ are the concentrations of the oxidized and reduced species, respectively.

- **Factors Affecting Redox Potential**:
  - **Concentration**: Changing the concentration of reactants or products alters the redox potential according to the Nernst equation.
  - **Temperature**: Redox potential is temperature-dependent due to its influence on reaction kinetics and equilibrium constants.
  - **pH**: The acidity or alkalinity of the solution affects redox potential by influencing the proton concentration and the distribution of species in solution.
  - **Pressure**: Pressure variations, though less common, can affect gas-phase reactions and thus redox potential.
  - **Nature of Electrodes**: Different reference electrodes can yield slightly different redox potentials due to differences in electrode materials and designs.

- **Applications**:
  - **Electrochemistry**: Redox potential is crucial in electrochemical processes such as batteries, fuel cells, and electrolysis.

- **Corrosion**: Understanding the redox potentials of metals and their environments helps predict and control corrosion processes.
- **Biochemistry**: Redox reactions play key roles in biological systems, including cellular respiration, photosynthesis, and antioxidant defense mechanisms.
- **Environmental Science**: Redox potential influences the fate and transport of pollutants in soils and water bodies.

- **Redox Couples**: In many redox reactions, substances exist in pairs called redox couples, consisting of an oxidized form and a reduced form. The standard redox potential of a redox couple is a characteristic property that depends on the specific chemical species involved.

Understanding redox potential is essential for predicting the direction and feasibility of redox reactions, as well as for designing and optimizing various chemical and electrochemical processes.

- **Define the following term**

Oxidation ,reduction, oxidizing agent ,reducing agent.

oxidation

Oxidation is a chemical reaction in which a substance loses electrons, leading to an increase in its oxidation state. It involves the addition of oxygen to a substance or the removal of hydrogen or electrons from it.

reduction.

Reduction is a chemical reaction in which a substance gains electrons, leading to a decrease in its oxidation state. It involves the addition of hydrogen or electrons to a substance or the removal of oxygen from it.

oxidizing agent.

An oxidizing agent is a substance that causes another substance to undergo oxidation by

accepting electrons in a chemical reaction. It is itself reduced in the process.

reducing agent.

reducing agent is a substance that causes another substance to undergo reduction by donating electrons in a chemical reaction. It is itself oxidized in the process.

(2marks)

- **Define cerimetry.**

Cerimetry: Cerimetry is a volumetric analytical technique used to determine the concentration of a substance in a sample by titrating it with a standard solution of cerium(IV) sulphate ($Ce(SO_4)_2$). The cerium (IV) ions oxidize the analyte, and the endpoint of the titration is detected using an indicator or by measuring a change in colour.

- **Define dichrometry.**

Dichrometry: Dichrometry is a quantitative analytical method based on the use of potassium dichromate ($K_2Cr_2O_7$) as a titrant. It is commonly employed for the determination of reducing agents in a sample. The dichromate ions undergo reduction in the presence of the analyte, and the endpoint is detected by a change in colour or using an indicator.

- **Differentiate oxidation and reduction.**

- **Oxidation vs. Reduction**:

    - Oxidation involves the loss of electrons from a chemical species, resulting in an increase in its oxidation state or a decrease in the number of its electrons.

    - Reduction involves the gain of electrons by a chemical species, leading to a decrease in its oxidation state or an increase in the

number of its electrons.

- In summary, oxidation is the process of losing electrons, while reduction is the process of gaining electrons.

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- **Differen**tiate iodometry and iodimetry.

- **Iodometry**: Iodometry is a volumetric titration method used to determine the concentration of oxidizing agents in a sample. It involves the addition of iodide ions (I^-) to the analyte, which is oxidized by the oxidizing agent. The endpoint of the titration is detected by the appearance of iodine, which can be starch-iodine complex or a colour change.

- **Iodimetry**: Iodimetry is a volumetric titration method used to determine the concentration of reducing agents in a sample. It involves the addition of iodine (I_2) to the analyte, which is reduced by the reducing agent. The endpoint of the titration is detected by the disappearance of iodine, often indicated by the decolorization of a starch-iodine complex or a color change.

MODEL QUESTION ANSWER FOR ELECTROCHEMICAL METHOD OF ANALYSIS

Unit -V

10 MARKS

- **explain the type of conductometric titration.**

Conductometric titration is a widely used analytical technique in pharmaceutical sciences for determining the concentration of various substances in solution. In conductometric titrations, the endpoint is detected based on changes in electrical conductivity as a titrant is added to a solution containing the analyte. Here's an explanation of the different types of conductometric titrations:

- **Strong Acid-Strong Base Titrations**:
    - In this type of titration, a strong acid (e.g., HCl) is titrated with a strong base (e.g., NaOH).
    - Initially, both the acid and base solutions conduct electricity well due to the presence of ions.
    - As the titration proceeds, the conductivity gradually decreases due to neutralization, until a sudden increase in conductivity occurs at the equivalence point when all the acid has reacted with the base.
    - The endpoint is detected by a sharp increase in conductivity, which can be observed using a conductometer.
- **Weak Acid-Strong Base Titrations**:
    - Here, a weak acid (e.g., acetic acid) is titrated with a strong base (e.g., NaOH).
    - Initially, the conductivity is low due to the weakly ionized acid.
    - As the strong base is added, it neutralizes the acid, resulting in an increase in conductivity.

    - The endpoint is determined by the inflection point on the titration curve, where the conductivity changes most rapidly.

- **Strong Acid-Weak Base Titrations**:

    - This type involves titrating a strong acid (e.g., HCl) with a weak base (e.g., NH_3).
    - Initially, the conductivity is high due to the strong acid.
    - As the weak base is added, it reacts with the acid, leading to a decrease in conductivity.
    - The endpoint is detected by a sudden drop in conductivity, corresponding to complete neutralization.

- **Redox Titrations**:

    - Conductometric titrations can also be used for redox reactions, where the endpoint is determined by a change in the conductivity resulting from the oxidation or reduction of the analyte.
    - For example, in titrations involving iodine (I_2) as the titrant and thiosulfate ($S_2O_3^{2-}$) as the analyte, the endpoint is reached when all the iodine has reacted with the thiosulfate, resulting in a sudden decrease in conductivity.

- **Complexometric Titrations**:

    - In complexometric titrations, conductivity changes occur due to the formation or dissociation of complexes between metal ions and chelating agents.
    - The endpoint is detected by a change in conductivity resulting from the formation or dissociation of the metal-ligand complexes.

- **Write a note on conductivity cell with application of conductometric titration.**

A conductivity cell is a crucial component in conductometric titration, a widely used analytical technique in pharmaceutical sciences for determining the concentration of substances in solution. This note elucidates the structure and function of a conductivity cell and its application in conductometric titration.

Structure of a Conductivity Cell: A conductivity cell typically comprises two electrodes immersed in the solution being analysed. These electrodes are connected to a conductivity meter, which measures the electrical conductivity of the solution. The two common types of conductivity cells are:

- Two-Electrode Cell:
    - Consists of two electrodes: a pair of metal electrodes (often platinum) immersed in the solution.
    - Measures the conductivity between these two electrodes.
- Four-Electrode Cell:
    - Consists of two pairs of electrodes: a pair for current injection and another pair for voltage measurement.
    - Allows for more accurate measurements by minimizing errors associated with electrode polarization.

Function of a Conductivity Cell: The conductivity cell functions by measuring the ability of a solution to conduct electricity. When an electric potential is applied across the electrodes, ions in the solution migrate towards oppositely charged electrodes, resulting in the flow of electric current. The conductivity meter measures this current, which is proportional to the solution's conductivity. Factors affecting conductivity include the concentration and mobility of ions in the solution.

Application in Conductometric Titration: Conductivity cells are extensively used in conductometric titrations, where the endpoint of a titration is determined by monitoring changes in electrical conductivity as titrant is added to the analyte solution. The

application of conductivity cells in conductometric titrations offers several advantages:

- Endpoint Detection:
    - In conductometric titrations, the endpoint is detected by a sudden change in conductivity, indicating the completion of the reaction between the analyte and titrant.
    - The conductivity cell allows for precise detection of this endpoint, leading to accurate determination of the analyte concentration.
- Wide Applicability:
    - Conductivity cells can be used for various types of titrations, including acid-base, redox, and complexometric titrations.
    - They are particularly useful for titrations involving strong acids, strong bases, and salts, where conductivity changes are significant.
- Rapid Analysis:
    - Conductometric titrations offer rapid analysis compared to other titration methods, making them suitable for high-throughput analysis in pharmaceutical laboratories.
- Sensitivity and Precision:
    - Conductivity cells offer high sensitivity and precision, allowing for the detection of small changes in conductivity and accurate determination of analyte concentrations.

Conclusion: Conductivity cells play a crucial role in conductometric titrations, enabling accurate and rapid determination of analyte concentrations in pharmaceutical analysis. Understanding the structure and function of conductivity cells is essential for pharmacy students to effectively utilize this technique in their analytical work.

- **Describe the theory of conductometric titration.**

The theory of conductometric titration is based on the principle of measuring the change in electrical conductivity of a solution as a titrant is added to the analyte solution. Conductometric titration is widely used in analytical chemistry for determining the concentration of various substances in solution. Here's a detailed explanation of the theory behind conductometric titration:

- **Principle of Conductivity**: Electrical conductivity is a measure of a solution's ability to conduct electricity. It depends on the presence and mobility of ions in the solution. Solutions containing ions (such as electrolytes) conduct electricity, while non-ionic solutions (such as pure water) have low conductivity.

- **Titration Process**:

    - In conductometric titration, a titrant solution of known concentration is gradually added to the analyte solution.

    - Initially, the analyte solution may have a different conductivity than the titrant solution.

    - As the titrant is added, a chemical reaction occurs between the analyte and titrant, resulting in the formation of a product.

- **Change in Conductivity**:

    - The addition of titrant leads to changes in the concentration of ions in the solution, affecting its conductivity.

    - At the beginning of the titration, the conductivity may remain relatively constant if the analyte and titrant solutions have similar conductivity.

    - As the titration proceeds, the conductivity of the solution changes due to the formation or consumption of ions during the chemical reaction.

- **Endpoint Detection**:

    - The endpoint of the titration is reached when the stoichiometric amount of titrant has reacted with the analyte.

- At the endpoint, there is a sudden change in conductivity, indicating that the reaction is complete.
- This change in conductivity can be detected using a conductometer, a device that measures the electrical conductivity of the solution.

- **Titration Curve**:
  - A plot of conductivity (or its reciprocal, resistance) against the volume of titrant added is known as a titration curve.
  - The titration curve typically shows a gradual change in conductivity until the endpoint is reached, where there is a sharp increase or decrease in conductivity, depending on the nature of the titration.

- **Types of Titration**:
  - Conductometric titration can be applied to various types of chemical reactions, including acid-base, redox, and complexometric titrations.
  - In acid-base titrations, the endpoint is reached when the pH of the solution undergoes a sudden change, which corresponds to a significant change in conductivity.
  - In redox titrations, the endpoint is detected by the change in conductivity resulting from the oxidation or reduction of the analyte.

In conclusion, conductometric titration relies on measuring changes in electrical conductivity to determine the endpoint of a titration. Understanding the theory behind conductometric titration is essential for accurate and precise analytical measurements in various fields, including pharmaceutical analysis, environmental monitoring, and quality control.

- **Explain the type of potentiometric titration.**

Potentiometric titration is a widely used analytical technique in which the endpoint of a titration is determined by measuring the potential difference (voltage) between two electrodes in a solution. This method is based on the principle that the concentration of an analyte can be determined by measuring its

ability to change the electrical potential of a solution. There are several types of potentiometric titrations, each suited for different types of chemical reactions and analytes. Here's an explanation of some common types:

- **Acid-Base Titration**:
    - In acid-base titrations, the titration involves the neutralization reaction between an acid and a base.
    - A pH electrode (glass electrode) is used to measure the change in pH of the solution as the titrant is added.
    - At the endpoint of the titration, the pH undergoes a sudden change, indicating the complete neutralization of the analyte.
    - Common indicators for acid-base titrations include phenolphthalein, methyl orange, and bromothymol blue. Potentiometric titration eliminates the need for indicators, offering greater precision and accuracy.
- **Redox Titration**:
    - Redox titrations involve oxidation-reduction reactions between the analyte and titrant.
    - A redox electrode, such as a platinum electrode or a combination electrode (e.g., platinum and Ag/AgCl reference electrode), is used to measure changes in the redox potential of the solution.
    - The endpoint of the titration is determined by a sudden change in the redox potential, indicating the completion of the redox reaction.
    - Common redox titrations include the determination of oxidizing agents (e.g., permanganate, iodine) or reducing agents (e.g., thiosulfate, ascorbic acid).
- **Complexometric Titration**:

- Complexometric titrations involve the formation of complex ions between metal ions and chelating agents (complexing agents).
- A metal or ion-selective electrode is used to measure changes in the concentration of free metal ions in solution.
- The endpoint of the titration is determined by a sudden change in the electrode potential, indicating the formation or dissociation of metal-ligand complexes.
- EDTA (ethylenediaminetetraacetic acid) is a commonly used chelating agent in complexometric titrations.

- **Precipitation Titration**:

  - Precipitation titrations involve the formation of a precipitate through a precipitation reaction between the analyte and titrant.
  - A combination electrode or a suitable ion-selective electrode is used to monitor changes in the concentration of ions in solution.
  - The endpoint is determined by a sudden change in the electrode potential, indicating the formation of a precipitate or the completion of the precipitation reaction.
  - Examples include the titration of chloride ions with silver nitrate or sulphate ions with barium chloride.

- potentiometric titration offers a versatile and precise method for determining the concentration of various analytes in solution. By utilizing different types of electrodes and monitoring changes in potential, potentiometric titrations can be applied to a wide range of chemical reactions and analytes, providing accurate and reliable results in analytical chemistry.

- **How end point is detected by potentiometric titration.**

In potentiometric titration, the endpoint is detected based on changes in the electrical potential (voltage) of the solution as titrant is added to the analyte solution. The endpoint is the point at which the stoichiometric amount of titrant has reacted with the analyte, indicating the completion of the titration. Here's how the endpoint is detected in potentiometric titration:

- **Selection of Electrodes**:
    - Potentiometric titration requires the use of suitable electrodes to measure changes in electrical potential. Common electrodes include glass electrodes (pH electrodes), redox electrodes (platinum or combination electrodes), metal ion-selective electrodes, and ion-selective electrodes specific to certain ions or species.
- **Initial Potential Measurement**:
    - Before the titration begins, the potential of the solution is measured using the selected electrode(s). This provides a baseline or starting point for the titration.
- **Titrant Addition**:
    - The titrant, usually a solution of known concentration, is gradually added to the analyte solution while stirring continuously. As the titrant reacts with the analyte, the composition of the solution changes, leading to changes in electrical potential.

- **Potential Monitoring**:

    - Throughout the titration, the potential of the solution is continuously monitored using the selected electrode(s). The potential is recorded at regular intervals or continuously, depending on the sensitivity of the equipment and the nature of the titration.

- **Detection of Inflection Point**:

    - As the titration progresses, the potential of the solution may change gradually until it reaches a point where it changes more rapidly. This point is known as the inflection point or equivalence point region.

    - At the inflection point, the rate of change in potential is at its maximum. It indicates that the amount of titrant added is close to the stoichiometrically equivalent amount required to react with the analyte.

- **Endpoint Determination**:

    - The endpoint of the titration is located close to the inflection point, but it may not always coincide exactly with it. The endpoint is determined by extrapolating or interpolating the data to identify the point where the potential undergoes a sudden, sharp change.

    - This sudden change in potential indicates that the titration is complete, and the endpoint has been reached.

- **Verification and Replication**:

- To ensure accuracy and reliability, the endpoint determination may be verified by repeating the titration or performing replicate titrations. Consistent results across multiple titrations confirm the accuracy of the endpoint determination.

The endpoint in potentiometric titration is detected by monitoring changes in electrical potential as titrant is added to the analyte solution. The endpoint is determined based on the inflection point or sudden change in potential, indicating the completion of the titration and the attainment of stoichiometric equivalence between the analyte and titrant.

- **What is PH how it can be determined? enlist types of electrodes.**

Potentiometric titration is a widely used analytical technique in which the endpoint of a titration is determined by measuring the potential difference (voltage) between two electrodes in a solution. This method is based on the principle that the concentration of an analyte can be determined by measuring its ability to change the electrical potential of a solution. There are several types of potentiometric titrations, each suited for different types of chemical reactions and analytes. Here's an explanation of some common types:

- **Acid-Base Titration**:

  - In acid-base titrations, the titration involves the neutralization reaction between an acid and a base.
  - A pH electrode (glass electrode) is used to measure the change in pH of the solution as the titrant is added.
  - At the endpoint of the titration, the pH undergoes a sudden change, indicating the complete neutralization of the analyte.
  - Common indicators for acid-base titrations include phenolphthalein, methyl orange, and bromothymol blue. Potentiometric titration eliminates the need

for indicators, offering greater precision and accuracy.

- **Redox Titration**:

    - Redox titrations involve oxidation-reduction reactions between the analyte and titrant.

    - A redox electrode, such as a platinum electrode or a combination electrode (e.g., platinum and Ag/AgCl reference electrode), is used to measure changes in the redox potential of the solution.

    - The endpoint of the titration is determined by a sudden change in the redox potential, indicating the completion of the redox reaction.

    - Common redox titrations include the determination of oxidizing agents (e.g., permanganate, iodine) or reducing agents (e.g., thiosulfate, ascorbic acid).

- **Complexometric Titration**:

    - Complexometric titrations involve the formation of complex ions between metal ions and chelating agents (complexing agents).

    - A metal or ion-selective electrode is used to measure changes in the concentration of free metal ions in solution.

    - The endpoint of the titration is determined by a sudden change in the electrode potential, indicating the formation or dissociation of metal-ligand complexes.

    - EDTA (ethylenediaminetetraacetic acid) is a commonly used chelating agent in complexometric titrations.

- **Precipitation Titration**:

    - Precipitation titrations involve the formation of a precipitate through a precipitation reaction between the analyte and titrant.

    - A combination electrode or a suitable ion-selective electrode is used to monitor changes in the concentration of ions in solution.

    - The endpoint is determined by a sudden change in the electrode potential, indicating the formation of a precipitate or the completion of the

precipitation reaction.

- Examples include the titration of chloride ions with silver nitrate or sulfate ions with barium chloride.

In summary, potentiometric titration offers a versatile and precise method for determining the concentration of various analytes in solution. By utilizing different types of electrodes and monitoring changes in potential, potentiometric titrations can be applied to a wide range of chemical reactions and analytes, providing accurate and reliable results in analytical chemistry.

In potentiometric titration, the endpoint is detected based on changes in the electrical potential (voltage) of the solution as titrant is added to the analyte solution. The endpoint is the point at which the stoichiometric amount of titrant has reacted with the analyte, indicating the completion of the titration. Here's how the endpoint is detected in potentiometric titration:

- **Selection of Electrodes**:
    - Potentiometric titration requires the use of suitable electrodes to measure changes in electrical potential. Common electrodes include glass electrodes (pH electrodes), redox electrodes (platinum or combination electrodes), metal ion-selective electrodes, and ion-selective electrodes specific to certain ions or species.
- **Initial Potential Measurement**:
    - Before the titration begins, the potential of the solution is measured using the selected electrode(s). This provides a baseline or starting point for the titration.
- **Titrant Addition**:
    - The titrant, usually a solution of known concentration, is gradually added to the analyte solution while stirring continuously. As the titrant reacts with the analyte, the composition of the solution changes, leading to changes in electrical potential.

- **Potential Monitoring**:
    - Throughout the titration, the potential of the solution is continuously monitored using the selected electrode(s). The potential is recorded at regular intervals or continuously, depending on the sensitivity of the equipment and the nature of the titration.
- **Detection of Inflection Point**:
    - As the titration progresses, the potential of the solution may change gradually until it reaches a point where it changes more rapidly. This point is known as the inflection point or equivalence point region.
    - At the inflection point, the rate of change in potential is at its maximum. It indicates that the amount of titrant added is close to the stoichiometrically equivalent amount required to react with the analyte.
- **Endpoint Determination**:
    - The endpoint of the titration is located close to the inflection point, but it may not always coincide exactly with it. The endpoint is determined by extrapolating or interpolating the data to identify the point where the potential undergoes a sudden, sharp change.
    - This sudden change in potential indicates that the titration is complete, and the endpoint has been reached.
- **Verification and Replication**:
    - To ensure accuracy and reliability, the endpoint determination may be verified by repeating the titration or performing replicate titrations. Consistent results across multiple titrations confirm the accuracy of the endpoint determination.

In summary, the endpoint in potentiometric titration is detected by monitoring changes in electrical potential as titrant is added to the analyte solution. The endpoint is determined based on the inflection point or sudden change in potential, indicating the completion of the titration and the attainment of stoichiometric equivalence between the analyte and titrant.

pH, or "potential of Hydrogen," is a measure of the acidity or alkalinity of a solution. It represents the concentration of hydrogen ions ($H+$) in a solution. pH is a logarithmic scale ranging from 0 to 14, where pH 7 is considered neutral, pH less than 7 indicates acidity, and pH greater than 7 indicates alkalinity.

The determination of pH in electrochemical analysis involves the use of electrodes that respond to changes in the concentration of hydrogen ions in the solution. These electrodes generate an electrical potential (voltage) proportional to the pH of the solution. There are several types of electrodes used for pH determination, each with its unique characteristics and applications:

- **Glass Electrode (pH Electrode)**:
    - The glass electrode is the most commonly used electrode for pH measurement.
    - It consists of a thin glass membrane that selectively interacts with hydrogen ions in the solution.
    - The potential across the glass membrane changes with the concentration of $H+$ ions, providing a direct measurement of pH.
    - The glass electrode is highly sensitive, precise, and suitable for a wide range of pH measurements.
- **Reference Electrode**:
    - A reference electrode is used in combination with a pH electrode to measure pH accurately.
    - The reference electrode provides a stable reference potential against which the potential of the pH electrode is measured.
    - Common reference electrodes include the Ag/AgCl electrode, calomel electrode (Hg/Hg2Cl2), and saturated calomel electrode (SCE).
- **Combination Electrode**:
    - Combination electrodes integrate both the pH electrode and reference

electrode into a single unit.

- This design simplifies pH measurement by eliminating the need for a separate reference electrode.
- Combination electrodes are convenient for routine pH measurements in laboratories and field applications.

- **Ion-Selective Electrodes (ISEs)**:
  - Ion-selective electrodes are designed to selectively respond to specific ions in solution, including hydrogen ions (pH).
  - The ion-selective membrane of these electrodes selectively interacts with the target ion, generating a potential that is proportional to its concentration.
  - ISEs are available for various ions, such as fluoride, chloride, sodium, potassium, and calcium ions.

- **Metal Oxide Electrodes**:
  - Metal oxide electrodes, such as the antimony electrode, respond to changes in the concentration of hydrogen ions.
  - These electrodes consist of a metal oxide layer that interacts with $H+H+$ ions, generating a potential that varies with pH.
  - Metal oxide electrodes are less common than glass electrodes but may be used in specific applications.

In electrochemical analysis, the selection of the appropriate electrode depends on factors such as the sample matrix, pH range, sensitivity requirements, and environmental conditions. By utilizing different types of electrodes, electrochemists can accurately determine pH and analyse various chemical systems with precision and reliability.

- **Explain dropping mercury electrode and rotating platinum electrode.**

Dropping Mercury Electrode (DME):

- **Introduction**:

    - The dropping mercury electrode (DME) is a type of working electrode used in electrochemical analysis.

    - It consists of a capillary tube filled with mercury that continuously replenishes a small droplet of mercury at the electrode surface.

- **Construction**:

    - The DME typically consists of a glass capillary tube attached to a reservoir containing mercury.

    - A small portion of the capillary tube is immersed in the electrolyte solution being analysed.

    - A constant stream of mercury flows from the reservoir through the capillary due to gravity or controlled by a syringe pump.

    - As the mercury droplet forms at the tip of the capillary, it serves as the working electrode surface.

- **Working Principle**:

    - The mercury droplet at the electrode surface provides a large active area for electrochemical reactions to occur.

    - Due to its liquid nature, the mercury droplet readily amalgamates with many metals, facilitating the study of metal ions in solution.

    - The mercury surface is continually renewed as fresh mercury droplets form, ensuring consistent electrode performance.

- **Applications**:

    - DME is commonly used in voltammetry techniques such as cyclic voltammetry, stripping voltammetry, and polarography.

    - It is particularly useful for the determination of trace metals and heavy

metal ions in solution due to its high sensitivity and wide potential range.

- DME is also employed in studies of organic compounds, electrode kinetics, and surface adsorption phenomena.

Rotating Platinum Electrode (RPE):

- **Introduction**:
    - The rotating platinum electrode (RPE) is a specialized electrode used in electrochemical studies, particularly in electroanalytical chemistry.
    - It consists of a platinum wire or disk electrode that rotates at a controlled speed during electrochemical measurements.
- **Construction**:
    - The RPE typically consists of a platinum wire or disk electrode mounted on a shaft connected to a motor.
    - The electrode may be polished to ensure a smooth and uniform surface for electrochemical reactions.
    - The rotation speed of the electrode is precisely controlled using a motor and speed controller.
- **Working Principle**:
    - The rotation of the platinum electrode enhances mass transport of reactants to and from the electrode surface.
    - As the electrode rotates, it creates a hydrodynamic flow in the solution, which promotes mixing and minimizes concentration gradients near the electrode surface.
    - This increased mass transport improves the reproducibility and sensitivity of electrochemical measurements.

- **Applications**:

    - RPE is widely used in techniques such as rotating disk electrode (RDE), rotating ring-disk electrode (RRDE), and rotating cylinder electrode (RCE).

    - RDE is commonly employed in studies of reaction kinetics, electrode mechanisms, and electrocatalysis.

    - RRDE allows for the investigation of both the primary reaction at the disk electrode and the secondary reactions occurring at the ring electrode simultaneously.

    - RCE is utilized in studies of electrodeposition, corrosion, and electrochemical surface modification processes.

Dropping mercury electrode (DME) and rotating platinum electrode (RPE) are two important types of working electrodes used in electrochemical analysis. While DME offers a continuously renewing mercury surface for sensitive measurements of metal ions, RPE enhances mass transport and improves the reproducibility of electrochemical measurements through controlled rotation. Both electrodes play crucial roles in various electroanalytical techniques and contribute to the advancement of electrochemical research.

- **Describe about polarography and explain ilkovic equation.**

Polarography is an electrochemical technique used for qualitative and quantitative analysis of substances in solution. It was developed by Jaroslav Heyrovský in the 1920s and has since become a valuable tool in analytical chemistry due to its simplicity, sensitivity, and versatility. Here's a detailed overview of polarography:

- **Principle of Polarography**:

    - Polarography is based on the principle of voltammetry, where the current passing through a solution is measured as a function of an applied voltage.

    - The technique typically employs a three-electrode system consisting of a working electrode (often a dropping mercury electrode), a reference electrode (e.g., Ag/AgCl), and an auxiliary or counter electrode (usually

platinum).

- **Electrode Setup**:

    - The working electrode, often made of mercury, is immersed in the solution being analysed. Mercury electrodes are chosen because they readily form amalgams with many metals, facilitating sensitive detection.

    - The reference electrode provides a stable reference potential against which the potential of the working electrode is measured.

    - The auxiliary or counter electrode completes the electrical circuit and ensures that the current passing through the solution is controlled by the potential applied to the working electrode.

- **Operation**:

    - A potential is applied between the working and reference electrodes, causing electrochemical reactions to occur at the surface of the working electrode.

    - As the potential is varied, electrochemical processes such as reduction or oxidation of analyte species take place.

    - The resulting current is measured using a sensitive ammeter or potentiated and plotted against the applied potential to generate a polarogram.

- **Polarogram**:

    - A polarogram is a plot of current (y-axis) versus applied potential (x-axis) obtained during polarography.

    - Peaks or waves in the polarogram correspond to different electrochemical processes occurring at the electrode surface.

    - The position and shape of these peaks provide information about the concentration, identity, and electrochemical behavior of substances in solution.

- **Applications**:

    - Polarography finds applications in various fields including environmental analysis, pharmaceuticals, food and beverage industries, and research.

    - It is particularly useful for the determination of trace metals, heavy metal ions, organic compounds, and redox-active species in solution.

    - Polarography is also employed in studies of electrode kinetics, reaction mechanisms, and surface adsorption phenomena.

- **Advantages**:

    - Polarography offers several advantages including high sensitivity, selectivity, and simplicity.

    - It requires minimal sample preparation and can be performed quickly and inexpensively.

    - The technique provides valuable insights into the electrochemical behavior of substances, making it a versatile tool in analytical chemistry.

polarography is a powerful electrochemical technique used for the analysis of substances in solution. Its ability to provide quantitative and qualitative information about analytes makes it indispensable in various fields of research and industry.

The Ilkovič equation is a fundamental equation used in polarography to describe the relationship between the current passing through the solution and the applied potential. It is named after the Slovak chemist Jaroslav Ilkovič, who developed it in the mid-20th century. The Ilkovič equation is particularly important in understanding the diffusion-controlled processes that occur during polarographic measurements.

The Ilkovič equation is expressed as:

$$i=k\cdot n\cdot A\cdot D1/2\cdot Ci=k\cdot n\cdot A\cdot D_{1/2}\cdot C$$

Where:

- ii is the current (in amperes, A).
- kk is a proportionality constant depending on the electrode surface area and other experimental parameters.
- nn is the number of electrons involved in the electrochemical reaction.
- AA is the electrode surface area (in cm^2).
- DD is the diffusion coefficient of the analyte (in cm^2/s).
- CC is the concentration of the analyte (in mol/cm^3).

The Ilkovič equation describes the diffusion-controlled current, where the current is proportional to the square root of the diffusion coefficient (DD) and the concentration (CC) of the analyte. Let's break down each component of the equation:

- **Proportionality Constant (kk)**:
    - The proportionality constant kk depends on various experimental factors, including the electrode surface area, the geometry of the electrochemical cell, and the properties of the solution.
    - It accounts for factors such as the efficiency of mass transfer to the electrode surface and the sensitivity of the detection system.
- **Number of Electrons (nn)**:
    - nn represents the number of electrons transferred during the electrochemical reaction occurring at the electrode surface.
    - It is determined by the stoichiometry of the reaction.
- **Electrode Surface Area (AA)**:
    - AA refers to the effective surface area of the working electrode.
    - It influences the rate of electrochemical reactions and the amount of analyte that can be detected.
- **Diffusion Coefficient (DD)**:
    - DD is a measure of the rate at which the analyte diffuses through the solution.

- It depends on the properties of the analyte, such as its size, shape, and viscosity of the solvent.
- Higher diffusion coefficients result in faster mass transport to the electrode surface, leading to higher currents.

- **Concentration of Analyte (C $\boldsymbol{C}$)**:
  - C $\boldsymbol{C}$ represents the concentration of the analyte in solution.
  - It influences the number of analyte molecules available for electrochemical reaction and, therefore, the current measured.

The Ilkovič equation provides valuable insights into the kinetics of electrochemical reactions occurring at the electrode surface during polarography. By understanding the relationship between current, potential, and experimental parameters, researchers can optimize polarographic measurements for accurate and precise determination of analyte concentrations.

- **Describe about polarography and its application.**

Polarography is an electrochemical technique used for qualitative and quantitative analysis of substances in solution. It was developed by Jaroslav Heyrovský in the 1920s and has since become a valuable tool in analytical chemistry due to its simplicity, sensitivity, and versatility. Here's a detailed overview of polarography:

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- The technique typically employs a three-electrode system consisting of a working electrode (often a dropping mercury electrode), a reference electrode (e.g., Ag/AgCl), and an auxiliary or counter electrode (usually platinum).

- **Electrode Setup**:

  - The working electrode, often made of mercury, is immersed in the solution being analysed. Mercury electrodes are chosen because they readily form amalgams with many metals, facilitating sensitive detection.

  - The reference electrode provides a stable reference potential against which the potential of the working electrode is measured.

  - The auxiliary or counter electrode completes the electrical circuit and ensures that the current passing through the solution is controlled by the potential applied to the working electrode.

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  - The position and shape of these peaks provide information about the concentration, identity, and electrochemical behavior of substances in

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    - It is particularly useful for the determination of trace metals, heavy metal ions, organic compounds, and redox-active species in solution.
    - Polarography is also employed in studies of electrode kinetics, reaction mechanisms, and surface adsorption phenomena.

- **Advantages**:

    - Polarography offers several advantages including high sensitivity, selectivity, and simplicity.
    - It requires minimal sample preparation and can be performed quickly and inexpensively.
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polarography is a powerful electrochemical technique used for the analysis of substances in solution. Its ability to provide quantitative and qualitative information about analytes makes it indispensable in various fields of research and industry.

(5 MARKS)

- **Write the construction and working of potentiometric titration.**

Potentiometric titration is a technique used in analytical chemistry to determine the concentration of a substance in a solution by measuring the potential difference between two electrodes immersed in the solution. Here's how it works:

Construction:

- **Electrodes:** The setup consists of a reference electrode and an indicator electrode. The reference electrode is usually a saturated calomel electrode (SCE) or a

silver/silver chloride electrode, which provides a stable reference potential. The indicator electrode is usually a glass electrode or a metal electrode specific to the analyte being titrated.

- **Burette:** A burette containing the titrant solution is connected to a dispensing system, allowing controlled delivery of the titrant into the sample solution.

- **Potentiometer:** A potentiometer or a pH meter is used to measure the potential difference between the reference and indicator electrodes. The potentiometer displays the potential difference as a function of the volume of titrant added.

Working:

- **Preparation:** The sample solution containing the analyte to be titrated is prepared in a suitable container. The reference electrode and the indicator electrode are immersed in the sample solution.

- **Initial Reading:** The initial potential (voltage) between the reference electrode and the indicator electrode is noted using the potentiometer.

- **Titration:** The titrant solution is added dropwise from the burette into the sample solution while stirring continuously. As the titrant reacts with the analyte, the concentration of ions in the solution changes, causing a change in potential at the indicator electrode.

- **Endpoint Detection:** The endpoint of the titration is detected by monitoring the potential difference using the potentiometer. The endpoint is typically reached when the potential difference exhibits a sudden change, indicating that the stoichiometric amount of titrant has reacted with the analyte.

- **Calculation:** The volume of titrant added at the endpoint is recorded. From the volume and concentration of the titrant solution, the concentration of the analyte in the sample solution can be calculated using stoichiometry.

Potentiometric titration is widely used in various applications, including acid-base titrations, redox titrations, and complexometric titrations, due to its high precision and accuracy.

- **describe the silver chloride electrode**

The silver chloride electrode is a type of reference electrode commonly used in electrochemical measurements and potentiometric titrations. It consists of a silver wire coated with silver chloride (AgCl) immersed in a saturated potassium chloride (KCl) solution. Here's a description of its construction and functioning:

Construction:

- **Silver Wire:** The electrode is typically made of a pure silver wire, which serves as the conductive element. This silver wire is usually coated with a layer of silver chloride.

- **Silver Chloride Coating:** The silver wire is coated with a layer of silver chloride (AgCl) through a process called chloritization. This layer of AgCl is crucial for establishing a stable and reproducible potential.

- **Saturated KCl Solution:** The electrode is immersed in a solution of potassium chloride (KCl) that is saturated with AgCl. This solution serves as the electrolyte, providing a medium for ion transport and maintaining a constant concentration of chloride ions.

Functioning:

- **Reference Potential:** The silver chloride electrode establishes a stable reference potential based on the redox reaction between silver ions (Ag^+) and chloride ions (Cl^-) according to the equation:

$$AgCl(s) \rightleftharpoons Ag+(aq)+Cl-(aq) AgCl(s) \rightleftharpoons Ag+(aq)+Cl-(aq)$$

This equilibrium ensures that there is a constant concentration of silver ions and chloride ions in the solution surrounding the electrode, leading to a stable reference potential.

- **Stability:** The presence of the AgCl layer ensures that the electrode potential remains constant over time, making it suitable for accurate and reproducible measurements.

- **Junction Potential:** To minimize the formation of a liquid junction potential, which could interfere with measurements, the junction between the saturated KCl solution and the sample solution is typically made with a ceramic frit or a salt bridge.

- **Reference Electrode:** The silver chloride electrode is used as a reference electrode in combination with another electrode (e.g., a working electrode or an indicator electrode) to measure potentials or perform electrochemical measurements in various applications, such as pH measurements, potentiometric titrations, and electrochemical studies.

The silver chloride electrode is widely used in electrochemical experiments and analytical chemistry due to its stability, reproducibility, and relatively low cost.

- **give basic principle of potentiometry.**

The basic principle of potentiometry revolves around measuring the potential difference (voltage) between two electrodes in a chemical system to determine the concentration of an analyte in a solution. Here's a breakdown of the basic principle:

- **Electrode Potential:** Each electrode in the system has a characteristic potential (voltage) relative to a reference electrode. This potential arises from the redox reactions occurring at the electrode-electrolyte interface. In potentiometry, one of the electrodes serves as a reference electrode with a known and stable potential.

- **Ion Selective Electrode:** The other electrode, known as the indicator electrode, is selectively sensitive to the analyte of interest. This electrode responds to changes in the concentration of the analyte by generating a potential difference.

- **Equilibrium Potential:** When the analyte is present in the solution, it establishes an equilibrium between its ionized and un-ionized forms. This equilibrium potential, governed by the Nernst equation, is directly proportional to the logarithm of the analyte concentration.

- **Measurement:** By measuring the potential difference between the reference electrode and the indicator electrode using a potentiometer or a pH meter, the concentration of the analyte can be determined based on the relationship between the electrode potential and the analyte concentration.

- **Endpoint Detection:** During titration or monitoring, the endpoint of the reaction or the change in analyte concentration is indicated by a sudden change in the potential difference between the electrodes. This change signifies that the stoichiometric amount of titrant has reacted with the analyte or that a specific condition has been reached.

- **Calibration:** Prior to measurements, calibration is performed to establish a relationship between the potential difference and the analyte concentration. This calibration curve allows for the accurate determination of unknown concentrations based on measured potentials.

Overall, potentiometry offers a simple, rapid, and precise method for quantitative analysis of various chemical species in solution, making it a widely used technique in analytical chemistry, environmental monitoring, pharmaceutical analysis, and many other fields.

- **write the principle of polarography.**

The principle of polarography revolves around the electrochemical reduction or oxidation of analytes at a dropping mercury electrode (DME) while applying a linearly changing potential. This technique is based on the concept that the current passing through the electrochemical cell is directly proportional to the concentration of the analyte being reduced or oxidized at the electrode surface.

Here's a concise breakdown:

- **Electrode Configuration**: Polarography typically employs a dropping mercury electrode (DME) as the working electrode, immersed in the solution containing the analyte. The DME continuously replenishes its mercury surface by dropping mercury from a reservoir, ensuring a fresh electrode surface for each measurement.
- **Potential Variation**: A potential is applied to the working electrode, which is then linearly varied over time. This potential variation enables the reduction or oxidation of the analyte species present in the solution.
- **Reduction/Oxidation Reactions**: As the potential changes, reduction or oxidation reactions occur at the surface of the mercury electrode. These reactions involve the transfer of electrons between the analyte species and the electrode, leading to the formation of reduced or oxidized products.
- **Current Measurement**: The current flowing through the electrochemical cell is measured as a function of the applied potential. This current is directly proportional to the concentration of the analyte undergoing reduction or oxidation at the electrode surface.
  Polarogram Construction: The measured current is plotted against the applied potential to generate a polarogram. The resulting curve typically exhibits characteristic features such as peaks or waves corresponding to different analyte species present in the solution
- **write the construction and working of DME.**

A Dropping Mercury Electrode (DME) is a type of working electrode commonly used in electrochemical studies, particularly in polarography and voltammetry. It consists of a mercury drop suspended from a capillary tube, which serves as both the working electrode and the source of the mercury for the electrochemical reactions. Here's the construction and working principle of a DME:

Construction:

- **Capillary Tube:** The DME is typically constructed using a fine glass capillary tube. The capillary tube is tapered at one end to form a fine tip.

- **Mercury Reservoir:** The wider end of the capillary tube serves as a reservoir for the mercury. Mercury is introduced into the capillary tube from this reservoir.

- **Mercury Drop:** When the capillary tube is inverted, a small portion of the

mercury from the reservoir forms a drop at the tip of the capillary due to its surface tension.

Working Principle:

- **Formation of Mercury Drop:** The DME is initially prepared by filling the capillary tube with mercury and then inverting it, allowing a small mercury drop to form at the tip of the capillary.

- **Electrochemical Reactions:** During electrochemical measurements, the mercury drop serves as the working electrode, where various electrochemical reactions take place.

- **Analyte Interaction:** The analyte of interest, typically present in solution, interacts with the mercury drop through processes such as adsorption, absorption, or chemical reaction.

- **Voltage Application:** A voltage or potential is applied between the DME and a reference electrode. This voltage drives the electrochemical reactions at the surface of the mercury drop.

- **Electrochemical Processes:** Depending on the specific experiment or technique being employed, different electrochemical processes can occur at the DME surface, including:

    - Oxidation or reduction of analyte species.
    - Generation of mercury ions from the mercury drop.
    - Deposition or stripping of metal ions on/from the mercury surface.
    - Adsorption or desorption of species onto/from the mercury drop.

- **Signal Detection:** The electrochemical processes occurring at the DME surface generate electrical currents or potentials that can be measured using suitable

instrumentation, such as a potentiostat or a voltammeter.

- **Applications:** DMEs find applications in various electrochemical studies, including the determination of metal ions, organic compounds, and redox reactions, due to their versatility, reproducibility, and sensitivity.

Overall, DMEs are valuable tools in electrochemical research and analysis, providing insights into the kinetics, mechanisms, and thermodynamics of electrochemical processes.

Describe application of conductometric titration.

Conductometric titration is a type of titration where the progress of the reaction between the analyte and titrant is monitored by measuring the electrical conductivity of the reaction mixture. This technique finds numerous applications in analytical chemistry due to its simplicity, sensitivity, and versatility. Here are some common applications of conductometric titration:

- **Acid-Base Titrations:** Conductometric titration is widely used for the determination of acids and bases. During the titration, the conductance changes as the titrant is added, reaching a maximum or minimum at the equivalence point. This method is particularly useful for the titration of weak acids or bases, where the pH change may be small and difficult to detect using traditional indicators.

- **Redox Titrations:** Conductometric titration can also be employed for the determination of reducing or oxidizing agents in solution. Redox reactions involving changes in oxidation states lead to changes in the conductivity of the solution. This makes conductometric titration suitable for the determination of substances like iron, chlorine, iodine, and other redox-active species.

- **Complexometric Titrations:** Complexometric titrations involve the formation of complexes between metal ions and ligands. Conductometric titration can be applied to determine the concentration of metal ions by titrating them with a solution containing a complexing agent (ligand). The formation or dissociation of the metal-ligand complexes affects the conductivity of the solution, allowing for the determination of metal ion concentrations.

- **Precipitation Titrations:** In precipitation titrations, a precipitating agent is added to the analyte solution to form an insoluble precipitate. Conductometric titration can be used to monitor the formation of the precipitate and its subsequent dissolution as the titrant is added. This method is often employed for the determination of halides, sulfates, and other ions that form insoluble salts.

- **Non-Aqueous Titrations:** Conductometric titration can also be performed in non-aqueous solvents, expanding its applicability to systems where water-based titration methods may not be suitable. Non-aqueous conductometric titrations are commonly used in organic synthesis and pharmaceutical analysis.

- **Titration of Weakly Ionizable Substances:** Conductometric titration is particularly useful for titrating substances with low ionization constants or weak acids/bases. Since conductivity measurements are independent of the nature of the species involved, conductometric titration can accurately determine the endpoint of such titrations.

Overall, conductometric titration offers a versatile and sensitive method for the determination of a wide range of analytes in various types of samples, making it a valuable tool in analytical chemistry laboratories.

(2 MARKS)

- **Define ohms law state its unit.**

- **Ohm's Law:** Ohm's Law states that the current flowing through a conductor between two points is directly proportional to the voltage across the two points, provided the temperature remains constant. Mathematically, it is expressed as $V=IRV=IR$, where VV is the voltage (potential difference) across the conductor in volts (V), II is the current flowing through the conductor in amperes (A), and RR is the resistance of the conductor in ohms ($\Omega\Omega$).
  Unit: The unit of resistance, according to Ohm's Law, is the ohm ($\Omega\Omega$).

- **What is equivalent conductance.**

Equivalent Conductance: Equivalent conductance (denoted by $\Lambda\Lambda$) is the conductance of an electrolyte that occupies a volume containing one equivalent weight of the electrolyte when dissolved in a specific volume of solvent. It is calculated by dividing the molar conductance ($\Lambda m\Lambda_m$) of the electrolyte by its molarity (cC). Mathematically, $\Lambda=\Lambda mc\Lambda=c\Lambda_m$.

Unit: The unit of equivalent conductance is siemens per meter (S/m) or mho per centimeter (mho/cm

- **Define conductometry.**

- **Conductometry:** Conductometry is a technique used to measure the electrical conductivity of a solution. It involves passing an electric current through the solution and measuring the resulting conductance, which is the reciprocal of resistance. Conductometry is commonly employed in analytical chemistry for titrations and monitoring chemical reactions, as conductivity changes can indicate the progress of a reaction or the presence of specific ions.

- **What is potentiometric titration**

- **Potentiometric Titration:** Potentiometric titration is a method of quantitative chemical analysis based on measuring the voltage or potential difference between two electrodes in a solution. During the titration process, the potential difference between the two electrodes is monitored as titrant is added to the analyte solution. The endpoint of the titration is determined by detecting a sudden change in potential, indicating that the stoichiometric amount of titrant has reacted with the analyte. This technique is widely used in various analytical procedures due to its precision and accuracy.

- **Why salt bridge is used in potentiometric titration.**

The salt bridge is used in potentiometric titration to maintain electrical neutrality in the half-cells of the electrochemical cell. It allows for the flow of ions between the two half-cells without mixing the solutions, thus preventing the development of a potential difference due to the accumulation of charges. This ensures accurate potential measurements during titration by stabilizing the reference electrode and maintaining a constant ionic environment in the half-cells.