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Diploma in Pharmacy 1st Year
Pharmaceutical Chemistry
Important Questions
Chapter 2 : Volumetric and Gravimetric Analysis

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Chapter 2

Volumetric and Gravimetric Analysis

IMPORTANT Questions

Q1. Write a short note on volumetric analysis and explain acid base titration

Ans.

- The quantitative analytical method is widely used is volumetric analysis
- It is defined as the method involved in measurement of the volume of a solution whose concentration is known
- This method is also applied to determine the concentration of analyte
- Volumetric analysis or titration is defined as the measurement of volume of second substance which combines with the first known concentration

Procedure for volumetric analysis

- The solution that needs to be analysed should have a specific weight in the sample of +/- 0.0001g of the material
- It is important to choose right kind of material which is to be analysed to obtain accurate results
- Preference should be given to a substance that reacts rapidly and completely to produce a complete solution
- The titration should be continued until the reaction is completed and the amount of reactant added should be exactly the amount required to complete the reaction
- A weighed amount of reagent should be taken and dissolve into a solution if the reagent or reactant is to be made into a standard solution so that it is in a definitive volume within a volumetric flask

Acid Base Titration

- ❖ In chemistry, acid – base titration is used for analysis the unknown organic compound concentration of an acid and base
- ❖ The principle of acid-base titration is based on the neutralisation reaction occurring between acid & base
- ❖ Phenolphthalein is the most commonly used indicator for acid-base titration
- ❖ Acid-Base reaction involve transfer of proton,
- ❖ Example :- Base accept proton from Acid
- ❖ $H^+ + OH^- \rightarrow H_2O$
- ❖ It is most common Neutralisation reaction
- ❖ At equivalent point, moles of H^+ are equal to the moles of OH^-

- ❖ During titration one reactant (mostly an acid) is added from the burette to the known volume of the other reactant (mostly base) in a conical flask to make the equivalence point (end point) of the titration and indicator is used
- ❖ There are 3 theories , explaining the concept of acids and bases
 - Arrhenius theory
 - Bronsted Lowry theory
 - Lewis theory

Arrhenius theory

- ◆ The most commonly used concept of acids and bases was developed by Svante Arrhenius in 1884 termed as Arrhenius theory
- ◆ According to this theory an acid is a substance which dissociates in Aqueous Solution produce hydrogen ion on other hand a base is a substance which dissolves in aqueous solution to produce hydroxyl ion (OH⁻)
- ◆ For example HCl is an Arrhenius acid $\text{HCl} \rightarrow \text{H}^+ + \text{Cl}^-$
- ◆ NaOH is an Arrhenius base $\text{NaOH} \rightarrow \text{OH}^- + \text{Na}^+$
- ◆ Arrhenius theory was the first scientific theory that had given definition for acid and base as well as classified them It is the simplest theory and is useful in case of aqueous solution

Bronsted Lowry theory

- ♣ In 1923 J.N Bronsted and J.M Lowry was introduced a new concept of acid and base
- ♣ According to the theory an acid is any mol or ion that can donate a proton (H⁺) and base is any mol or ion that can accept a proton H⁺

Lewis theory

- ♠ This method of Acid & Base was given by G.N Lewis in the early 1930
- ♠ He defined Acid is an electron pair acceptor
 - Base is an electron pair donor
- ♠ In this theory the Lewis acid & Lewis base share an electron pair given by base result in the formation of a covalent or coordinate bond between them
- ♠ This resultant compound bounded with a covalent bond is known as a complex
- ♠ $A + B \rightarrow A-B$
- ♠ LA + LB → Complex
- ♠ According to this concept
- ♠ Lewis base are anion or molecule having a lone pair of electron
- ♠ Lewis Acid are cation or molecule lacking of electron pair

Q2. What is Complexometric titration discuss its principle & application

Ans. Complexometric titration

Complexometric titration chelatometry is a form of volumetric analysis in which the formation of a coloured complex is used to indicate the end point of a titration

Cl- are particularly useful for the determination of a mixture of different metal ions in solution

Principle

- ❑ In complexometric titration the metal ions are titrated with a complexing or a chelating agent
- ❑ This method is an analytical application of a complexation reaction
- ❑ This method involves transforming simple ion into a complex ions and determining the equivalence point using metal indicator or electrometrically
- ❑ This method is also termed as chelometric titration, chelometry titration, chelometric titration and EDTA titration Ethylene Diamine Tetra Acetic Acid

Application of complexometric titration

- Determination of permanent and temporary hardness of water separately
- Determination of total hardness of water
- Determination of magnesium and silicon dioxide in magnesium trisilicate
- Determination of calcium and lead in a mixture
- Determination of chromium III and Iron III in mixture kinetic masking
- Determination of manganese in the presence of Ironferromanganese
- Determination of lead and tin in a mixture
- Determination of phosphates

Q3. Write principle and application of gravimetric analysis

Ans. Gravimetric Analysis

- A quantitative analysis involving weight is known as gravimetric Analysis
- In which the substance to be analysed is covered into an insoluble precipitate
- This precipitate is collected, weighted using suitable method
- Precipitation is the most suitable technique which employs formulation of a precipitate not soluble in the solution

Examples

Analyte	Precipitant
Piperazine adopted tablets BP	picric acid
Piperazine phosphate BP	picric acid

Principle

- ❑ Law of mass action and reversible reaction

- ❑ Principle of solubility product
- ❑ Common ion effect

Law of mass action and reversible reaction

- According to the law of mass action the rate of reaction is directly proportional to the product molecular concentration of the reacting substance

Principle of solubility product

- ♣ The solubility product expression for a compound is the product of the concentration of its constituent ions each raised to the power that corresponds to the number of ions in one formula unit of the compound
- ♣ The quantity is constant at constant temperature for a saturated solution of the compound
- ♣ The statement is the solubility product principle
- ♣ A slightly soluble Salt is decreased if excess of either of its ions are added

Common ion effect

- ♠ The electron to the decrease in solubility of an ionic precipitation by the addition to the solution of a soluble compound with an ion in common with the precipitate

Types of Gravimetric Analysis

- I. **Physical Gravimetric**
- II. **Thermogravimetric**
- III. **Electro deposition :**
- IV. **Precipitative Gravimetry :**

Application

- Analysis of standard This is required during testing or instrument calibration
- Analysis Requiring Accuracy This analysis can be conducted using gravimetry Which being time consuming allows only a few determinations
- Pharmacopoeial analysis
- Lead as chromate This method has less application due to insolubility of chromates
- However This method helps in gaining experience in gravimetric Analysis
- The best results are obtained by precipitating from homogeneous solution using the homogeneous generation of chromate ions produced by slow oxidation of chromium (III) by bromate at 90- 95°C in the presence of an ethanoate buffer

Q4. Explain the non-Aqueous titration with principle and Applications

Ans. Non – Aqueous titration

- Titration involving very weak acid or base with the help of non-aqueous solvents to obtain sharp end point are non-aqueous titration

Principle

- ♦ Organic acids & bases are water insoluble very weak & cannot be analysed via conventional titration method thus non-aqueous titration is used which relies on the principle that non-aqueous solvents are used to dissolve the sample

Application

- ❖ The application of Non-Aqueous titration are
- ❖ Percentage of purity is determined by the assays
- ❖ Determination of Hydrophobic compounds
- ❖ Determination of the steroids
- ❖ Determination of Anti Tubercular drugs
- ❖ Determination of phenobarbitone

Q5. Write the principle and application of precipitation titration.

Ans. Precipitation titration

- ♣ Precipitation titration is a type of titration which involves the formation of precipitate during the titration techniques
- ♣ In precipitation titration the titrant reacts with analytic and forms an insoluble substance called precipitate
- ♣ For example AgNO_3 is used as a precipitating agent for the determination of Cl^-
- ♣ **Principle**
- ♣ Formation of an insoluble product by the combination of two ionic species is known as precipitation
- ♣ Precipitation reaction are not frequently used in titration because of the precipitation reaction do not comply with desired specification

Application of precipitation titration

- Precipitation reaction are applicable in removal of salts from water during water treatment in qualitative inorganic analysis and also in manufacturing of pigments
- Products of any reaction can also be isolated during work up by the precipitation reaction
- Precipitation reaction are also used in metallurgy

Q6. Give the Brief explanation of Redox titration.

Ans. Redox Titration

- ♠ In titration the redox/ oxidation- reduction reaction are more extensively used for analysis as compared to the precipitation reaction and acid base reaction
- ♠ Reduction is defined as the gain of one or more electron by atomic species or molecules
- ♠ Oxidation is loss of one or more electron by the atomic species or molecules oxidation is also known as de electronation
- ♠ A titration which deals with a reaction involving oxidation and reduction of certain chemicals species are known as redox Titration

Agents

Reducing agent : Sodium thiosulphate, ferrous sulphate, titanous sulphate, oxalic acid are some of the reducing agents

Oxidising agents: Potassium dichromate, potassium bromate etc are some of the oxidising agents

Types of Redox Titration

Based on the method

- **Direct titration :** In this method initially coloured substance are used Thus end point detection does not require an indicator
- **Back titration :** In this method the sample solution is titrated with excess volume of titrant solution

Application of Redox Titration

- ❖ Determination of phenol
- ❖ Determination of the presence of Iron in limonite
- ❖ Determination of calcium in lime stone
- ❖ Analysis of Isoniazid
- ❖ Analysis of tocophenol