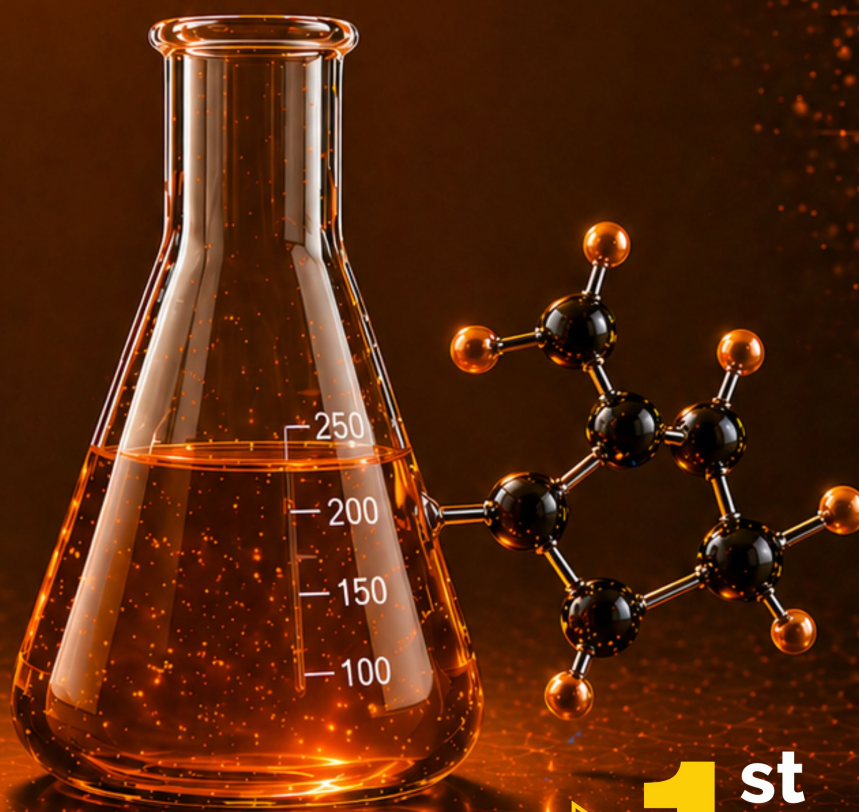


A Text Book of
**PHARMACEUTICAL INORGANIC
AND ANALYTICAL CHEMISTRY**

As per the New PCI Syllabus **NEP 2020**

BP106T



1st SEM.

Bachelor of Pharmacy



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BP106T

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PREFACE

We are delighted to present this book, Pharmaceutical Analysis and Inorganic Chemistry, prepared in accordance with the latest Pharmacy Council of India (PCI) syllabus and the vision of the National Education Policy (NEP) 2020 for First Year B.Pharm (Semester I) students. NEP 2020 promotes holistic, multidisciplinary, learner-centric, and outcome-based education with emphasis on conceptual understanding, critical thinking, problem-solving, skill development, and lifelong learning. Keeping these objectives in mind, this book has been designed to strengthen the fundamental concepts of pharmaceutical analysis and inorganic pharmaceutical chemistry while encouraging analytical and practical learning.

Pharmaceutical Analysis and Inorganic Chemistry form the foundation of pharmaceutical sciences by enabling students to understand the identity, purity, quality, and safety of drugs and pharmaceutical substances. The knowledge gained from these subjects is essential for laboratory practice, quality control, quality assurance, pharmaceutical manufacturing, research, regulatory affairs, and clinical applications. A strong understanding of these disciplines equips students with the scientific skills and analytical approach required for higher education, competitive examinations, and successful careers in the pharmaceutical industry.

The book is systematically organized into five units covering pharmaceutical analysis, analytical errors, standards, impurities and limit tests; acid-base chemistry, buffer systems, isotonicity, and electrolytes; volumetric and gravimetric analysis; gastrointestinal agents and radiopharmaceuticals; and miscellaneous inorganic pharmaceutical compounds with their pharmaceutical applications. The content has been presented in a logical sequence using simple language, well-labelled illustrations, solved examples, and review questions to promote conceptual clarity, self-learning, and competency-based education.

We sincerely hope that this book will serve as a valuable resource for students, teachers, and pharmacy professionals. We welcome constructive suggestions from readers, which will help us improve future editions and continue contributing to quality pharmacy education.

Authors

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Note

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Unit I

Introduction to Pharmaceutical Analysis, Errors, Impurities



Syllabus

Introduction to pharmaceutical analysis

- Different techniques of analysis, Methods of expressing strength of solutions, Primary and secondary standards with examples.

Errors

- Sources of errors, types of errors, methods of minimizing errors, accuracy, precision and significant figures.

Impurities

- Definition, types, contents and regulatory importance. Sources and types of impurities in Pharmaceuticals, limit tests for chloride, sulphate, iron, arsenic, lead, heavy metals, and modified limit test for chloride and sulphate.



INTRODUCTION TO PHARMACEUTICAL ANALYSIS, ERRORS, IMPURITIES



LEARNING OBJECTIVES

After studying this chapter, the learner will be able to:

- Understand the principles and importance of pharmaceutical analysis.
- Differentiate between analytical techniques and standards used in pharmaceutical testing.
- Explain sources of errors and apply measures to improve accuracy and precision.
- Understand the concept, sources, and regulatory importance of pharmaceutical impurities.
- Identify and perform limit tests for common impurities such as chloride, sulphate, iron, arsenic, lead, and heavy metals.
- Apply significant figures and solution strength calculations in pharmaceutical analysis.

1.1 INTRODUCTION TO PHARMACEUTICAL ANALYSIS

- Pharmaceutical analysis is a branch of chemistry that applies **analytical methods** to ensure the **purity, quality, and safety** of **pharmaceutical products**.
- It includes both **qualitative and quantitative analysis**, involving a series of procedures for **identification, measurement, quantification, and purification** of **substances**.
- The main applications of pharmaceutical analysis include identifying the chemical nature of compounds, determining components in mixtures, separating mixture constituents, and isolating, purifying, and structurally characterizing **compounds**.

❑ Scope of Pharmaceutical Analysis

1. Drug Development & Research

Used to identify and characterize APIs and new drug molecules. Spectroscopy and mass spectrometry help determine structure, purity, and efficacy.

2. Quality Control (QC) & Quality Assurance (QA)

QC detects defects and ensures product quality through testing. QA focuses on preventing defects by maintaining and improving manufacturing processes.

3. Impurity Profiling

Detects and quantifies impurities, residual solvents, and contaminants. Ensures drug safety, quality, and regulatory compliance.

4. Clinical & Disease Diagnostics

Analyzes biomarkers, proteins, and biological fluids for disease detection. Supports accurate diagnosis and treatment planning.

5. Pharmacopoeial Compliance

Verifies that pharmaceutical products meet standards of pharmacopoeias such as IP, USP, and BP. Ensures legal and regulatory acceptance.

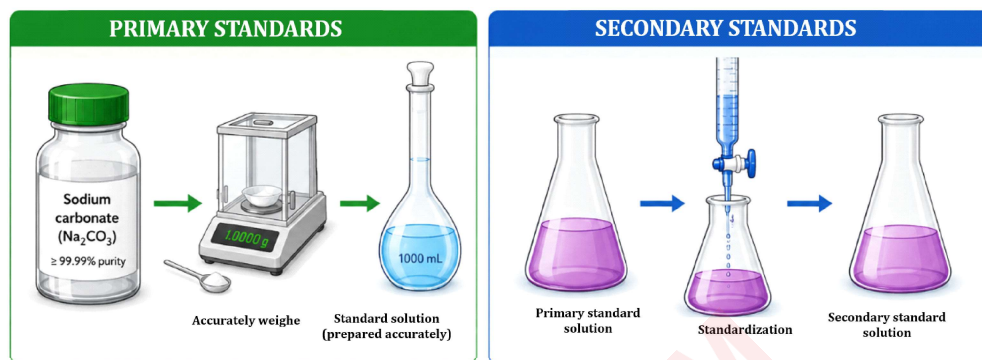


Fig 1.12:- Primary and Secondary Standard Substances

1.2 ERRORS

- The term error refers to the **difference between the measured and true value** in the results of any analytical operation.
- The error of a measurement is an inverse measure of its accuracy.
- The smaller the error, the greater the accuracy of the analysis.
- Errors can be expressed as either Absolute error or Relative error.

Absolute error = Observed value - True value

Relative error = $\frac{(\text{Observed value} - \text{True value})}{(\text{True value})} \times 100\%$

1.2.1 Sources of errors

- Errors in the results of an analysis can result from various sources.
 - Some major sources of errors in pharmaceutical analysis are described hereunder.
- 1. Human Source**
 - ✓ The qualification and experience of the analyst have a major impact on the accuracy.
 - ✓ Experiments performed by inexperienced persons have a higher chance of error compared to those performed by experienced analysts
 - 2. Instrumental, Apparatus, and Glassware**
 - ✓ Use of low-quality instruments, glassware, and apparatus increases the chances of error significantly.
 - ✓ Uncalibrated instruments further contribute to inaccurate results.
 - 3. Experimental Conditions**
 - ✓ Carrying out analysis under unfavourable or unsuitable conditions for a particular experiment leads to undesirable results.
 - 4. Constituents Used in Analysis**
 - ✓ Use of standards, solvents, reagents, and other constituents that are not of the required quality and purity results in error outcomes.
 - 5. Procedure**
 - ✓ Using an analytical procedure that is not validated increases the risk of errors.
 - ✓ Even a validated procedure, if not followed carefully, can lead to inaccurate results.

4. **Sensitivity of a Limit Test:** Since most limit tests are carried out using dilute solutions and the results depend upon the concentration of the trace impurity present, it may take a longer time for the results to become visible or appreciable. Therefore, the duration of the test is a very important factor that must be carefully considered while designing a limit test.

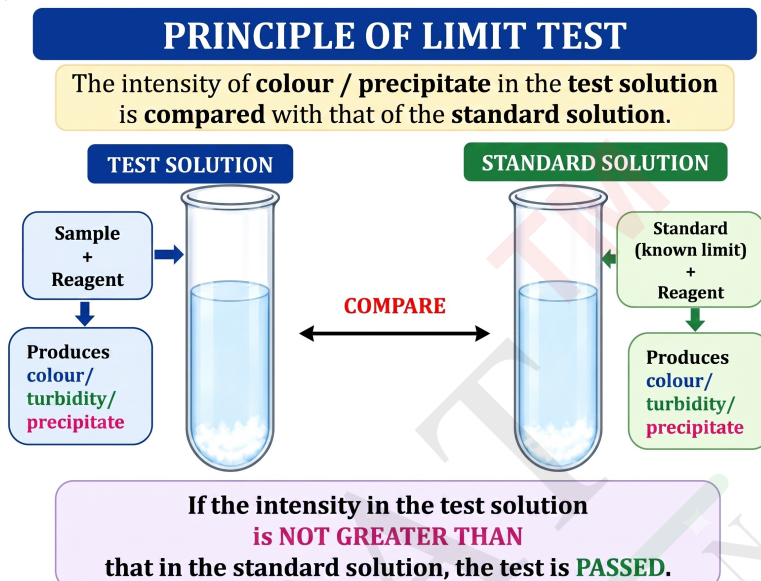


Fig 1.18:- General Principle of Limit Test

1.3.5 Limit tests for chloride

- Principle**

- ✓ The limit test for chloride is based upon the **reaction of chloride** impurities with **silver nitrate** in the **presence of dilute nitric acid**.
- ✓ The turbidity/opalescence produced in the test solution is compared with the standard turbidity/opalescence produced or obtained by the reaction of a known quantity of chloride with silver nitrate.

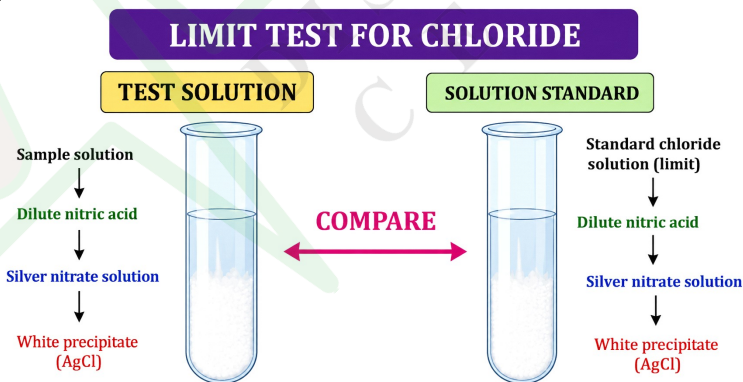
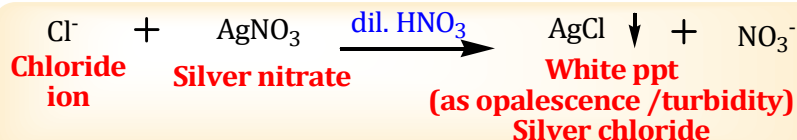


Fig 1.19: Limit test for Chloride



- ✓ The silver chloride precipitate formed is insoluble in dilute nitric acid, which gives turbidity/opalescence.

- Reasons**

- ✓ Dilute nitric acid is used to dissolve other impurities, if present.

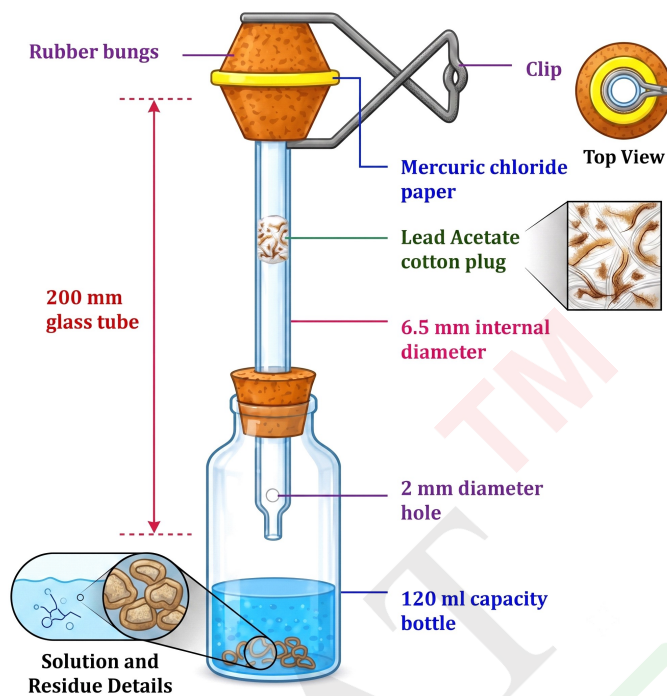


Fig 1.22: Gutzeit Apparatus for Limit Test of Arsenic

➤ Gutzeit Apparatus

- It consists of a wide-mouthed bottle (100 mL) fitted with a stopper.
- The stopper has a central hole through which a glass tube (8 mm external, 6.5 mm internal diameter, 200 mm length) is inserted.
- The upper end of the tube is fire-polished and flush with the stopper; the lower end is narrowed (1 mm opening) with a side hole (2 mm).
- The lower end and side hole remain below the stopper but above the 70 mL liquid level.
- The narrow end allows condensed vapours to return to the bottle without blocking the side hole.
- A cotton plug soaked in lead acetate is placed inside the tube (~25 mm below the top) to filter vapours.
- A dried filter paper dipped in mercuric chloride is placed on top of the stopper and covered with another rubber bung (6.5 mm hole aligned with the tube).
- Both stoppers are secured with spring clips.
- Due to this assembly, the liberated gas rises from the wide-mouthed bottle to the filter paper through the filter paper, cotton wool in that order.

• Procedure

Table 1.12 Procedure for limit test for Sulphate

S.No	Test Stain	Standard Stain
1.	Place the required amount of sample solution in the wide-mouthed bottle. Add 1 g potassium iodide + 10 g granulated zinc (AsT grade). Immediately close the bottle with the stopper assembly	Standard stain is prepared by using a dilute arsenic solution (0.132 gm of arsenic in 100 mL).
2.	To this, add 1 gm of potassium iodide and 10 gm of granulated zinc (AsT grade).	One mL of the above solution is diluted to 100 mL. This solution is transferred into a wide-mouthed bottle, and the remaining procedure is similar to that of the test.



ASSESS YOURSELF



Multiple Choice Questions

- Which of the following best defines pharmaceutical analysis
 - Study of drug metabolism in the body
 - Application of analytical techniques to identify and quantify drugs
 - Manufacturing process of tablets
 - Study of drug pharmacology
- Which of the following is a titrimetric method of analysis
 - UV spectrophotometry
 - Acid-base titration
 - HPLC
 - Flame photometry
- Which technique uses the principle of absorption of electromagnetic radiation by a substance
 - Gravimetry
 - Titrimetry
 - Spectrophotometry
 - Potentiometry
- Gravimetric analysis involves measuring
 - Volume of a solution
 - Mass/weight of the analyte
 - Electrical potential
 - Light absorption
- Which of the following is an example of an electroanalytical technique
 - TLC
 - Potentiometry
 - Fluorimetry
 - Karl Fischer titration
- What does 'w/v' mean in expressing solution strength
 - Weight of solvent per volume of solute
 - Weight of solute per volume of solution
 - Weight of solute per weight of solution
 - Volume of solute per volume of solvent
- A 1 M solution of NaOH (mol. wt. 40) contains how many grams per litre
 - 20 g/l
 - 40 g/l
 - 80 g/l
 - 10 g/l
- Normality is defined as
 - Moles of solute per litre
 - Equivalent weights of solute per litre
 - Grams of solute per 100 mL
 - Moles of solute per kg of solvent
- Parts per million (ppm) is used to express
 - Very concentrated solutions
 - Very dilute solutions
 - Normal solutions
 - Percentage solutions
- A 10% w/w solution means
 - 10 g solute in 100 mL solution
 - 10 g solute in 90 g solvent
 - 10 g solute in 100 g solution
 - 10 ml solute in 100 ml solution
- Molality is expressed in units of
 - mol/l
 - mol/kg of solvent
 - g/l
 - Equivalents/l
- Which of the following is a primary standard
 - NaOH
 - KMnO₄
 - Potassium hydrogen phthalate (KHP)
 - HCl
- A primary standard must NOT be
 - High purity
 - Stable under storage
 - Hygroscopic
 - High equivalent weight
- NaOH is classified as a secondary standard because
 - It is insoluble in water
 - It absorbs CO₂ and moisture from air
 - It reacts with glass
 - It has a low molecular weight
- Which of the following is used to standardise NaOH solution
 - HCl
 - Na₂CO₃
 - Oxalic acid
 - Potassium dichromate
- Standardisation of a secondary standard solution means
 - Preparing it freshly every day
 - Determining its exact concentration against a primary standard
 - Storing it in the dark
 - Filtering it before use

ANSWER KEY

1-b	2-b	3-c	4-b	5-b	6-b	7-b	8-b	9-b	10-c
11-b	12-c	13-c	14-b	15-c	16-b	17-c	18-c	19-c	20-b
21-c	22-b	23-c	24-b	25-b	26-c	27-c	28-c	29-b	30-b
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41-b	42-b	43-c	44-a	45-a	46-c	47-d	48-d	49-c	50-b

Short Answer Questions

1. Define pharmaceutical analysis and state its importance in the pharmaceutical industry.
2. Differentiate between qualitative and quantitative analysis with one example each.
3. What is volumetric analysis? Give two examples of volumetric methods.
4. Define molarity and molality. How do they differ.
5. What does "5% w/v" mean? Give one pharmaceutical example.
6. Calculate the normality of a solution containing 4 g of NaOH (M.W. = 40) in 500 ml.
7. Distinguish between w/w and w/v with examples.
8. Define a primary standard and list four essential properties it must possess.
9. Why is NaOH considered a secondary standard? How is it standardised.
10. Define accuracy and precision. How are they different.
11. What are systematic errors? Give two examples.
12. How can random errors be minimised in pharmaceutical analysis.
13. State the rules for determining significant figures and apply them to 0.00250.
14. What is meant by "running a blank" and what type of error does it correct.
15. Define impurities in pharmaceuticals and state their regulatory significance.
16. Classify impurities as per ICH guidelines with examples of each type.
17. State the difference between determinate and indeterminate errors with one example each.
18. Write a comprehensive note on the regulatory importance of impurity testing.
19. Write a short note on limit test for iron as per IP. State the principle, chemical reaction and reagents used.
20. Describe the limit test for chloride and also explain the modified limit test for chloride.

Long Answer Questions

1. Describe in detail the different techniques used in pharmaceutical analysis. Classify them with suitable examples.
2. Explain the various methods of expressing the strength of solutions with formulas and suitable pharmaceutical examples.
3. Define primary and secondary standards. Describe essential characteristics of a primary standard and give three examples each.
4. Discuss in detail the sources of errors in pharmaceutical analysis. Classify errors with examples and describe methods to minimise each type.
5. Define accuracy and precision with examples. Explain absolute error vs relative error. What are significant figures? State rules with examples.
6. Define impurities in pharmaceuticals. Classify them as per ICH guidelines and describe sources of each type with examples.
7. Describe the limit test for sulphate as per IP. Also explain the modified limit tests and why modifications are necessary.
8. Write in detail the limit tests for lead as per IP. Include reagents, principle, and procedure.
9. Describe the limit test for heavy metals as per IP. State the principle, reagents, and how results are compared.
10. With a neat labelled diagram, explain the Gutzeit apparatus used in the limit test for arsenic. Describe the principle, reagents, procedure, and interpretation of results as per IP.

Unit II

Acid-Base Chemistry and Buffer Systems in Pharmacy and Major extra and intracellular electrolytes



Syllabus

Acid-Base Chemistry and Buffer Systems in Pharmacy

- Definition of acids, bases, buffers, pH Scale and its significance, Buffer equation, calculation of pH for Buffer solution. Isotonicity and its application in IV Fluids and Ophthalmic Solutions.

Major extra and intracellular electrolytes

- Functions of major physiological ions, Electrolytes used in the replacement therapy: Sodium chloride*, Potassium chloride, Calcium chloride and Oral Rehydration Salt (ORS), Physiological acid base balance.



ACID-BASE CHEMISTRY AND BUFFER SYSTEMS IN PHARMACY AND MAJOR EXTRA AND INTRACELLULAR ELECTROLYTES



LEARNING OBJECTIVES

After studying this chapter, the learner will be able to:

- Understand the basic definitions of acids, bases and buffers and learn their importance in pharmaceutical sciences.
- Learn the concept of pH scale and understand how it affects biological systems, drug stability and drug absorption.
- Understand the Henderson-Hasselbalch equation and use it to calculate the pH of acidic and basic buffer solutions.
- Learn the concept of isotonicity and how it is applied in the preparation of IV fluids and ophthalmic solutions.
- Identify the major electrolytes present inside and outside the cells and understand their important physiological roles.
- Learn how Sodium Chloride, Potassium Chloride, Calcium Chloride and ORS are used in electrolyte replacement therapy.
- Understand the concept of acid-base balance in the body and know what happens when this balance is disturbed.

2.1 ACID, BASE CHEMISTRY AND BUFFER SYSTEMS IN PHARMACY

2.1.1 Definition of acids and bases

Table 2.1: Definition of acids and bases

ACIDS	BASES
• Acids are substances that produce hydrogen ions (H^+) when dissolved in water. 	• Bases are substances that liberate hydroxide ions (OH^-) when dissolved in water. 
• They have a pH less than 7, turn blue litmus paper red, possess a sour taste and react with bases to form salts and water. 	• They have a pH greater than 7, turn red litmus paper blue, exhibit a bitter taste and react with acids to produce salts and water. 
• Examples: Hydrochloric Acid, Boric Acid, Citric Acid and Acetylsalicylic Acid.	• Examples: Sodium hydroxide, Calcium hydroxide, Magnesium hydroxide and Potassium oxide.

2.1.3 pH SCALE

- The pH scale helps us **measure the acidity, basicity or neutrality** of a solution on a **scale of 0 to 14**, with 7 being the neutral marker; a pH above 7 is basic, and a pH below 7 is acidic.
- The pH scale was invented in 1909 by Danish biochemist **Soren Peter Lauritz Sorensen**.
- The pH of a solution equals the negative of the logarithm to the **base 10 of its hydrogen ion concentration, i.e., $\text{pH} = -\log_{10} [\text{H}^+]$** .

❖ pOH

- pOH is used as a measure of the **concentration of hydroxide ions (OH^-)**
- By definition, pOH is the negative logarithm (to the base 10) of the hydroxide ion concentration (mol/L).
- The concentration of hydroxide ions in water is related to the concentration of hydrogen cations by

$$[\text{OH}^-] = \frac{K_w}{[\text{H}^+]}$$

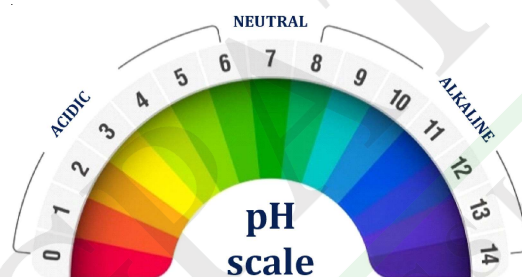
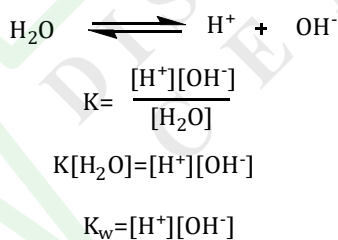


Fig. 2.1: pH Scale

❖ Hydrogen ion concentration, pH and pOH

Pure water ionizes as follows:



Where K_w is called ionic product of water and its value is 1×10^{-14} at 25°C , i.e.,

$$1 \times 10^{-14} = [\text{H}^+][\text{OH}^-]$$

Pure water has equal concentration of $[\text{H}^+][\text{OH}^-]$ ions. Thus

$$[\text{H}^+][\text{H}^+] = 10^{-14}$$

$$\text{or } [\text{H}^+] = 10^{-7}$$

- For **pure water**, the **concentration of hydrogen ions $[\text{H}^+]$ is 1×10^{-7} g-ion per litre**.
- A solution is considered **acidic** when the **$[\text{H}^+]$ concentration is greater than** and alkaline if **$[\text{H}^+]$ is less than g-ion per litre**.
- Conversely, a solution becomes **alkaline** when the hydroxide ion concentration **$[\text{OH}^-]$ exceeds 1×10^{-7} g**
- Therefore, if the concentration of either **$[\text{H}^+]$ or $[\text{OH}^-]$** is known, the other can be calculated using $1 \times 10^{-14} = [\text{H}^+][\text{OH}^-]$.

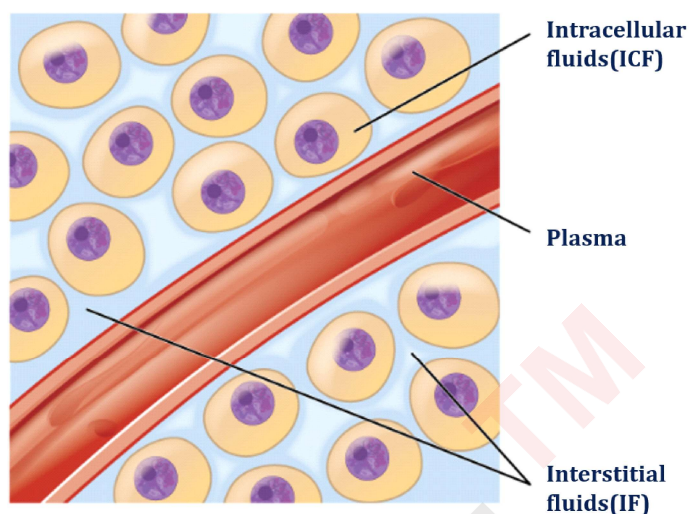


Fig. 2.2: Body fluid Compartment

1. Intracellular Body Fluid (ICF)

- ICF is the fluid formed inside the plasma membrane. It constitutes 45-50% of total body weight.
- It serves as the medium in which all the cell organelles are suspended.
- Potassium, Magnesium and Phosphate are the major ions in the ICF.

2. Extracellular Body Fluid (ECF)

- ECF includes interstitial and plasma (vascular) fluids. It constitutes 16-20% of the total body weight.
- It serves as a medium for the movement of ions, proteins and other nutrients.
- Sodium, chloride, calcium and bicarbonate are the major extracellular ions.

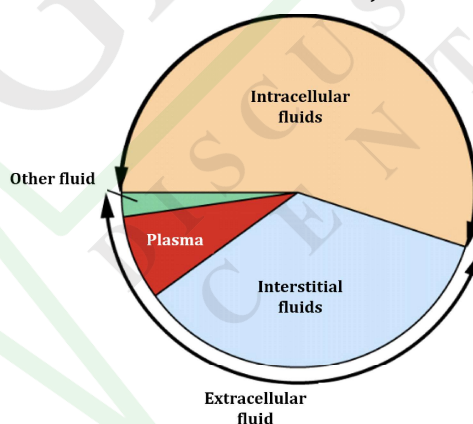


Fig. 2.3: Intra and Extra cellular fluids

Table 2.7: Major intra and extra cellular electrolytes

Electrolyte	Extracellular fluid (meq/L)		Intracellular fluid (meq/L)
	Plasma fluid	Interstitial fluid	
Cations			
Sodium (Na ⁺)	142	145	10
Potassium (K ⁺)	4.2	4	160
Calcium (Ca ²⁺)	5	3	10 ⁴ times < plasma level
Magnesium (Mg ²⁺)	2	2	35
Anions			
Chlorides (Cl ⁻)	108	117	4
Bicarbonates (HCO ³⁻)	24	27	10

- ✓ Bicarbonate buffer ($\text{HCO}_3^- / \text{H}_2\text{CO}_3$) in plasma and kidneys
- ✓ Phosphate buffer ($\text{HPO}_4^{2-} / \text{H}_2\text{PO}_4^-$) in kidney cells
- ✓ Haemoglobin buffer in RBCs
- These buffers are essential for normal cellular function.
- Excess H^+ ions are removed by three mechanisms:
 1. Buffer systems
 2. Exhalation of CO_2 (lungs)
 3. Renal excretion of H^+ (Kidneys)

❖ Buffer Systems

- Buffers are made of a weak acid and its corresponding base.
- They help maintain stable pH by quickly converting strong acids and bases into weaker forms.
- **Strong acids release more H^+ ions and decrease pH more, while strong bases increase pH more than weak bases.**

Major buffer systems in the body include:

- Protein buffer system
- Phosphate buffer system
- Carbonic acid–bicarbonate buffer system

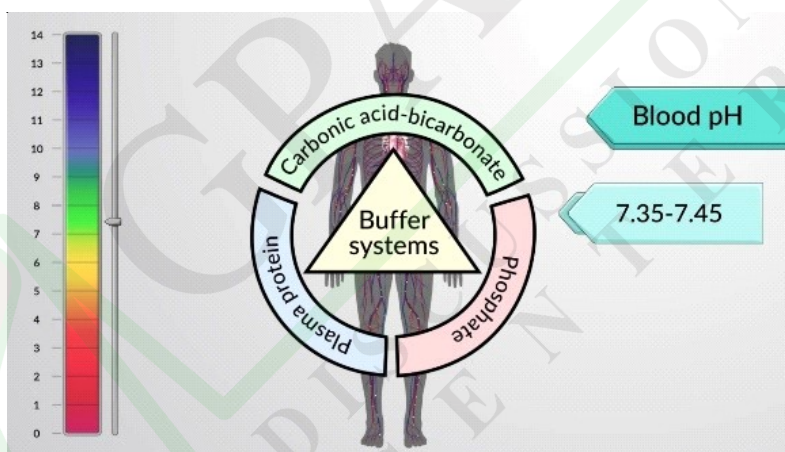


Fig. 2.4: Major buffer system in body

❖ Exhalation of CO_2

- Breathing plays a key role in regulating the pH of body fluids.
- **An increase in CO_2 levels raises H^+ concentration, thereby lowering pH (acidosis), while a decrease in CO_2 reduces H^+ and increases pH (alkalosis).**
- Increased ventilation removes more CO_2 , decreases H^+ concentration and results in a rise in blood pH.

❖ Renal Excretion of H^+

- Metabolism produces non-volatile acids (*e.g.*, sulphuric acid), which are eliminated by excreting H^+ in urine. Failure to remove these acids can lead to **acid–base imbalance and renal failure**.
- In the kidneys, cells of the proximal convoluted tubule (PCT) and collecting ducts secrete H^+ into the tubular fluid. In the PCT, Na^+ / H^+ exchangers help in H^+ secretion, while intercalated cells use proton pumps for this process.
- Some intercalated cells also have $\text{Cl}^- / \text{HCO}_3^-$ exchangers, facilitating the breakdown of carbonic acid into H^+ and HCO_3^- . The bicarbonate is reabsorbed into the blood, helping maintain pH balance.

45. The equation to calculate buffer capacity is

- (a) $\beta = \Delta pH / \Delta B$ (b) $\beta = \Delta H / \Delta pH$
 (c) $\beta = \Delta B / \Delta pH$ (d) $\beta = \Delta B / \Delta pB$

46. pH is equal to pka at

- (a) Half neutralization point
 (b) pH 14
 (c) pH 7
 (d) The neutralization point

47. In presence of Hydrofluoric acid, Nitric acid acts as

- (a) Lowry-Bronsted acid
 (b) Lowry-Bronsted base
 (c) Arrhenius acid

- (d) Can't be estimated

48. Normal saline contains

- (a) 0.9% w/v of sodium chloride
 (b) 0.9% w/w of sodium chloride
 (c) 9% w/v of sodium chloride
 (d) 5% w/v of sodium chloride

49. What volume of ORS is recommended after each loose stool for a child below 2 years of age

- (a) 40ml-55 mL (b) 100ml-150 mL
 (c) 10ml-30 mL (d) 50ml-100 mL

50. ORS stands for

- (a) Oral resolution Salts (b) Oral Rehydration Salts
 (c) Oral Restricted Salts (d) Oral Reserved solution

ANSWER KEY

1-c	2-b	3-c	4-a	5-b	6-b	7-c	8-b	9-b	10-c
11-c	12-b	13-b	14-c	15-a	16-b	17-b	18-b	19-b	20-b
21-b	22-a	23-b	24-c	25-c	26-b	27-d	28-b	29-b	30-d
31-c	32-c	33-b	34-c	35-a	36-a	37-b	38-d	39-d	40-a
41-b	42-a	43-c	44-c	45-c	46-a	47-a	48-a	49-d	50-b

Short Answer Questions

1. Define Arrhenius, Bronsted-Lowry, and Lewis's theories of acids and bases with one example each.
2. What is a conjugate acid-base pair? Give two examples from pharmaceutical systems.
3. Calculate the pH of a 0.01 M HCl solution and a 0.01 M NaOH solution.
4. What is the significance of pH in pharmaceutical formulations? Give two practical examples.
5. Define buffer solution and buffer capacity. What factors affect buffer capacity.
6. Derive the Henderson-Hasselbalch equation for an acidic buffer.
7. What are pharmaceutical applications of buffers? Give three examples.
8. Define isotonic, hypotonic, and hypertonic solutions. What happens to RBCs in each.
9. Why is isotonicity important in ophthalmic solutions? How is it adjusted.
10. List the major extracellular and intracellular electrolytes with their normal physiological concentrations.
11. Write a short note on sodium chloride as an electrolyte in replacement therapy.
12. What are the uses, dose, and precautions of potassium chloride in replacement therapy.
13. What is the role of calcium in the body? When is calcium chloride used therapeutically.
14. Write the composition of WHO-ORS and explain its mechanism of action.
15. What is physiological acid-base balance? Name the four types of acid-base disorders.
16. Define acidosis and alkalosis. What is the role of the kidneys in maintaining acid-base balance.
17. Compare the bicarbonate buffer system and phosphate buffer system in the body.
18. Explain the ionic product of water (K_w) and its significance in pH calculations.
19. Differentiate between hard acids/bases and soft acids/bases with suitable examples.
20. Discuss the electrolytes used in the replacement therapy.

Acid-base, Non-aqueous, Precipitation, Complexometric, Redox Titrations And Gravimetry

Unit III



Syllabus

Acid base titrations

- Theories of acid base indicators, classification of acid base titrations. Preparation and standardization of titrants viz. hydrochloric acid and sodium hydroxide. Theory involved in titrations of strong, weak, and very weak acids and bases, neutralization curves. Assay of Ammonium hydroxide.

Non-aqueous titrations

- Types of solvents used, acidimetric and alkalimetric titration using non aqueous solvents. Preparation and standardization of acidic and basic titrants. Estimation of weakly acidic and basic substances using non-aqueous titrants, estimation of Sodium benzoate.

Precipitation titrations and gravimetry

- Principle and steps involved in gravimetric analysis, Mohr's method, Volhard's, Modified Volhard's, Fajans method. Estimation of barium sulphate by gravimetry.

Complexometric titrations

- Classification, metal ion indicators, masking and demasking reagents, preparation and standardization of disodium EDTA. Estimation of Magnesium sulphate and Calcium gluconate*.

Redox titrations

- Concepts of oxidation and reduction, Types of redox titrations viz. Permanganometry, Cerimetry, Iodimetry, Iodometry and titrations with potassium iodate.



ACID-BASE, NON-AQUEOUS, PRECIPITATION, COMPLEXOMETRIC, REDOX TITRATIONS AND GRAVIMETRY

LEARNING OBJECTIVES

After studying this chapter, the learner will be able to:

- Understand the principles and classification of **acid-base titrations** and **acid-base indicators**.
- Explain **neutralization reactions**, titration curves, and the preparation, standardization, and assay of acid-base titrants.
- Describe the principles of **non-aqueous titrations**, types of solvents, and estimation of weak acids and bases.
- Understand the principles and applications of **precipitation titrations** and **gravimetric analysis**.
- Explain the concepts of **complexometric titrations**, EDTA standardization, metal ion indicators, and masking/demasking agents.
- Understand the principles of **redox titrations** and their pharmaceutical applications, including permanganometry, cerimetry, iodimetry, iodometry, and potassium iodate titrations.
- Apply volumetric and gravimetric analytical methods for the **assay of pharmaceutical substances**.

TITRATION

❖ Introduction of Titration

- **Titration** is a quantitative analytical method used to **determine the concentration of an unknown solution** by reacting it with a **standard solution of known concentration**.
- The process involves the gradual addition of one solution to another until the reaction reaches the **equivalence point or endpoint**.
- The **equivalence point** is typically detected using a **visual indicator that changes color** or *via* an **instrumental method**.

❖ Important Terminologies Used in Titration

1. **Titrant:** A reagent of known concentration and volume, often placed in a burette.
2. **Analyte:** The solution of unknown concentration.
3. **Equivalence Point:** The point at which the stoichiometric amount of titrant added equals the amount of analyte.

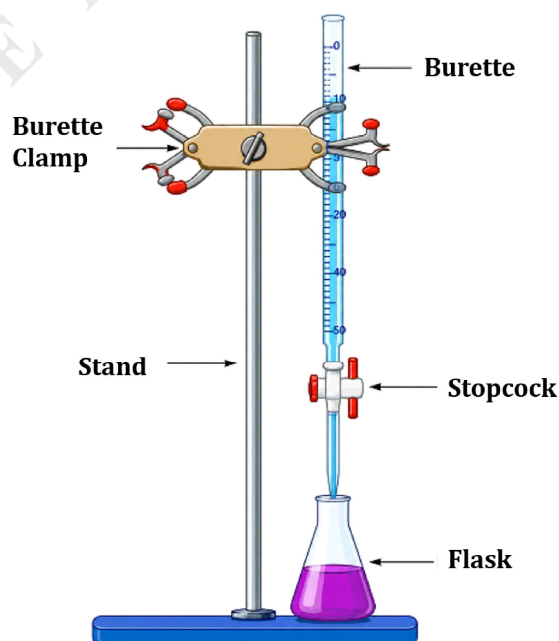
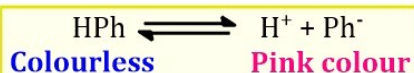


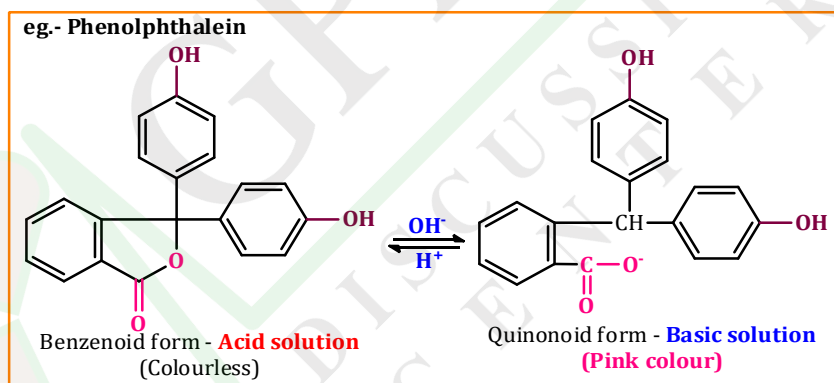
Fig. 3.1: Titration (Apparatus and Glassware)



- The undissociated molecules of phenolphthalein are colourless, whereas its ionized form (Ph^- ions) is pink in colour.
- In acidic solution, the high concentration of H^+ ions suppresses the ionization of phenolphthalein, so the solution remains colourless.
- When an alkali is added, OH^- ions combine with H^+ ions to form water, causing the equilibrium to shift towards ionization.
- As a result, more Ph^- ions are formed, producing a pink colour in the solution.

□ Resonance theory/ Quinonoid theory

- According to this theory, acid-base indicators are present in two tautomeric forms with different structural arrangements.
- These two forms remain in equilibrium with each other and are known as the benzenoid form and the quinonoid form.
- Both forms exhibit different colours, and the colour change occurs due to the conversion of one tautomeric form into the other.
- During a titration, the medium changes from acidic to alkaline or from alkaline to acidic.
- This change in pH shifts the equilibrium between the two forms, resulting in a visible colour change.



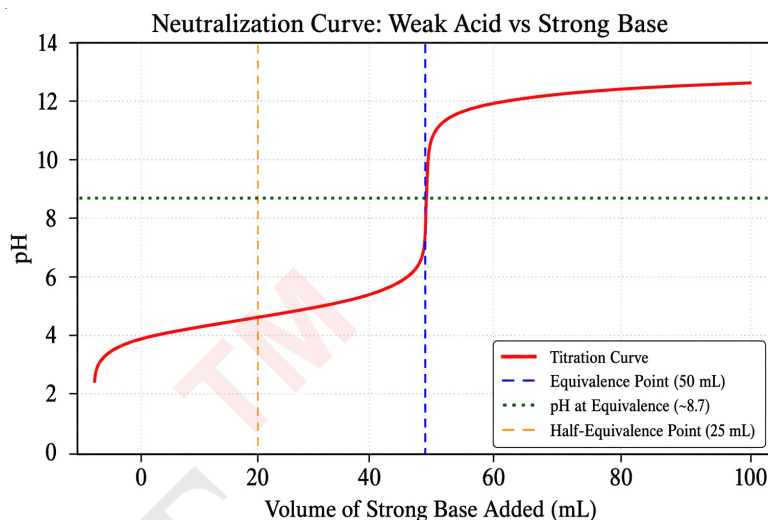
❖ Indicators of Acid-Base Titration

Table 3.3: Indicators used in acid -base titration

INDICATOR	pH RANGE	COLOUR ON ACIDIC SIDE	COLOUR ON BASIC SIDE
Methyl Violet	0.0 to 1.6	Yellow	Blue
Thymol blue	1.2-2.8	Blue	Yellow
Bromophenol Blue	3.0 to 4.6	Yellow	Blue
Methyl orange	3.1 to 4.4	Red	Orange
Methyl red	4.4 to 6.2	Red	Yellow
Bromothymol blue	6.0 to 7.6	Yellow	Blue
Phenol red	6.8 to 8.4	Yellow	Red
Cresol red	7.2 to 8.8	Yellow	Red
Phenolphthalein	8.3 to 10.0	Colourless	Pink

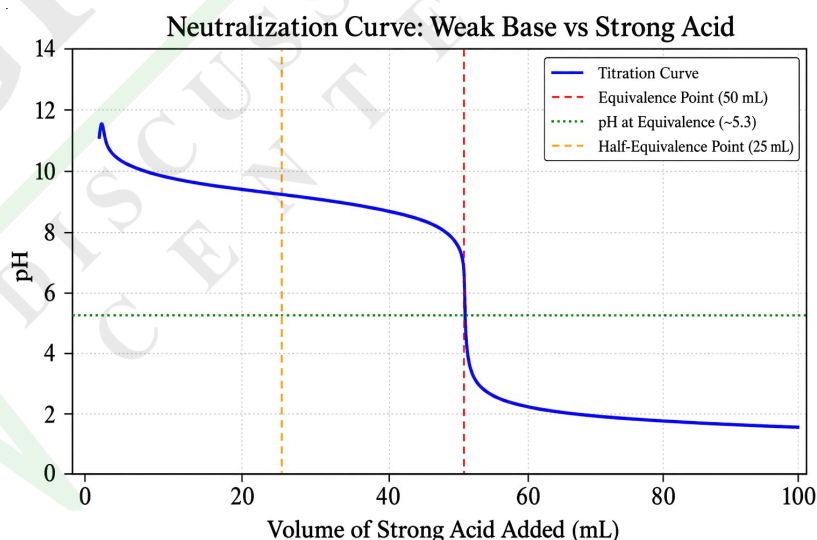
Weak acid-strong base

- In this titration, a weak acid such as acetic acid is titrated with a strong base like NaOH using a suitable indicator.
- Since the weak acid dissociates only partially, the initial pH is higher than that of a strong acid. As NaOH is added, the pH increases gradually.
- At the half-neutralization point, half of the weak acid is converted into its conjugate base, forming a buffer solution.
- At this stage: $\text{pH} = \text{pK}_a$
- In the buffer region, the pH changes only slightly even after adding more base.
- Near the equivalence point, the pH rises sharply because most of the weak acid has been neutralized.
- The equivalence point occurs above pH 7 due to the presence of the conjugate base formed during the reaction.
- After the equivalence point, excess NaOH causes a further increase in pH.



Weak base-strong acid

- In this titration, a weak base is titrated with a strong acid. Initially, the curve shows a high pH due to the presence of the weak base.
- As the strong acid is added, the pH decreases gradually because the weak base reacts slowly with the acid.
- At the equivalence point, all the weak base molecules are completely neutralized by the strong acid, causing a sharp fall in pH.
- The equivalence point occurs below pH 7 due to the formation of the conjugate acid of the weak base.
- After the equivalence point, the solution contains excess H^+ ions from the strong acid, so the pH is mainly controlled by the excess acid present.
- The hydrolysis of the salt formed is suppressed, and the curve continues to decrease gradually.



Weak base-weak acid

- A weak acid-weak base titration involves the reaction between a weak acid and a weak base.
- Since both substances ionize only partially, the pH changes very gradually during the titration.

$$N_1 V_1 = N_2 V_2$$

- Accurately weigh about 0.25 g of sodium benzoate and dissolve it in 20 mL of glacial acetic acid. Warm the solution to about 50°C, if necessary, then cool it.
- Titrate this solution with standardized 0.1 N perchloric acid using 2 drops of acetous crystal violet indicator.
- The appearance of a bluish-green colour marks the end point.

❖ Calculation

Each mL of 0.1 N HClO_4 = 0.01441 gm of $\text{C}_7\text{H}_5\text{NaO}_2$

$$\% \text{ Purity of } \text{C}_7\text{H}_5\text{NaO}_2 = \frac{\text{Volume of } \text{HClO}_4 \text{ (mL)} \times \text{Normality} \times 0.01441 \times 100}{\text{Weight of } \text{C}_7\text{H}_5\text{NaO}_2 \text{ (g)} \times 0.1}$$

❖ Result

- The percentage purity of the given sample of sodium benzoate was found to be x %.

3.3 PRECIPITATION TITRATIONS

□ Introduction

- Precipitation titration is a volumetric analytical method in which the titrant reacts with the analyte to produce an insoluble precipitate.
- The end point of the titration is identified when the analyte is completely consumed.
- This technique is commonly used for the determination of halide ions such as chloride (Cl^-), bromide (Br^-) and iodide (I^-) using silver nitrate.

❖ Argentometric Titrations

- Argentometric titration is a type of precipitation titration that involves the use of silver ions.
- In this method, silver nitrate (AgNO_3) is used as the precipitating agent to carry out the titration reaction.

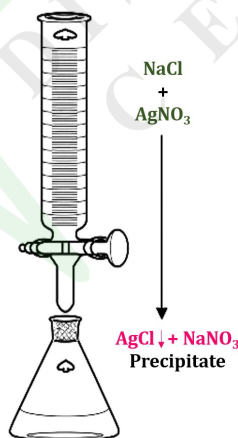
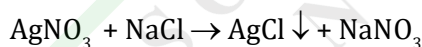


Fig. 3.4: Precipitation titration

3.3.1 Mohr's method

- Mohr's method is a precipitation titration used to determine the concentration of chloride ions in a solution by titrating it with silver nitrate solution.
- During the titration, silver nitrate is added gradually to the chloride-containing solution, resulting in the formation of a white precipitate of silver chloride.

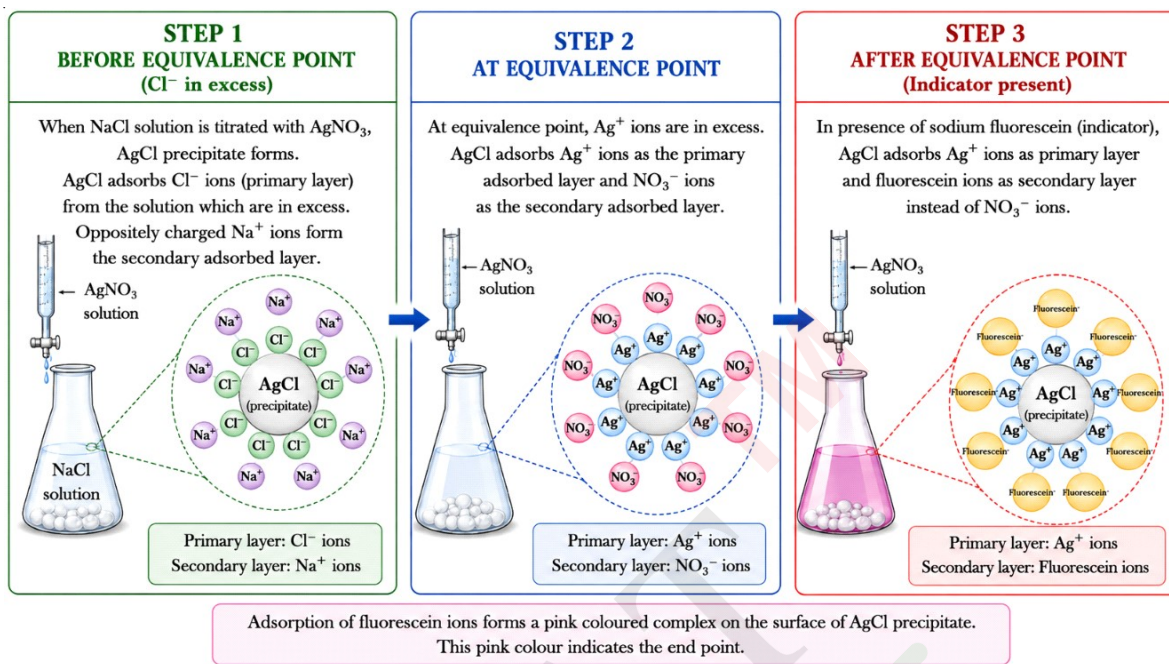


Fig. 3.7: Fajan's Method

3.4 GRAVIMETRIC ANALYSIS

Introduction

- Gravimetric analysis is a quantitative method in Analytical Chemistry used to determine the amount or concentration of a substance in a sample by measuring its mass.
- In this method, the analyte (substance being analyzed) is converted into an insoluble solid precipitate, which is then separated, washed, dried and weighed.
- The method is a quantitative analysis method by weight.
- From the weight of the compound, the calculation is done to determine the content.
- There are different ways through which the separation can be achieved which are given below.

1. Precipitation method (Precipitation gravimetry)

2. Volatilization or evolution method (Volatilization gravimetry)

3. Electro-analytical method (Electrogravimetry)

1. Precipitation Gravimetry

- In this method, the substance to be analyzed (analyte) is converted into an insoluble solid called a precipitate.
- The precipitate is separated by filtration, washed to remove impurities, dried or heated to obtain a stable compound and then weighed.
- The amount of analyte is calculated from the mass of the precipitate.

2. Volatilization Gravimetry

- In volatilization gravimetry, the sample is heated so that a volatile substance changes into vapor at a specific temperature.
- The vapor may be collected and weighed. If the analyte itself is volatile, its amount is determined from the decrease in the sample's mass after heating.

3. Electrogravimetry

- In electrogravimetry, the analyte is separated by depositing it on an electrode using electric current.

Mordant Black II	6 to 7	Red to Blue	Ca, Ba, Mg, Zn, Cd, Mn, Pb, Hg
Eriochrome Black T	6 to 7	Red to Blue	
Solochrome black T	6 to 7	Red to Blue	
Catechol Violet	8 to 10	Violet to red	Mn, Mg, Co, Zn
Thymol blue	10 to 12	Blue to gray	Pb, Zn, Cd, Hg
Calcon mixture	12 -14	Purple red	CaCO ₃ and CaCl ₂

❖ Instrumental Methods of Detection of End Point

❑ Spectrophotometric detection

- Spectrophotometric detection is based on the change in absorption spectrum that occurs when a metal ion forms a complex or when one complex changes into another. It provides more accurate endpoint detection than visual methods, especially in very dilute solutions.
- In EDTA titrations, accurate endpoints can be obtained even with 0.001 M solutions. Usually, indicators showing colour changes in the visible region are used, but coloured metal ions can also be titrated without indicators using spectrophotometric methods.
- It is also possible to detect end points in the ultraviolet region for colourless ions and complexes.

❑ Amperometric Titration

- Amperometric titration is based on measuring the diffusion current produced by metal ions at a selected half-wave potential. During complex formation with EDTA, the half-wave potential of the metal ion shifts to a more negative value.
- When EDTA is added gradually, the diffusion current decreases continuously as free metal ions are complexed. The endpoint is reached when the current becomes equal to the residual current, indicating complete complex formation.
- The volume of EDTA used at this point corresponds to the amount of metal ion present in the solution.

❑ Potentiometric Titration

- Potentiometric titration involves the use of a platinum electrode along with a standard reference electrode to measure the redox potential during complex formation. The platinum electrode detects the potential change associated with the metal-EDTA complex.
- Since disodium EDTA reacts more readily with metal ions in higher oxidation states, the redox potential decreases during the titration. The endpoint is determined from the sudden change in electrode potential.

3.5.3 Masking and demasking reagents

❖ Masking reagents

- Masking reagents form stable complexes with interfering metal ions without reacting with the metal ion being analyzed.

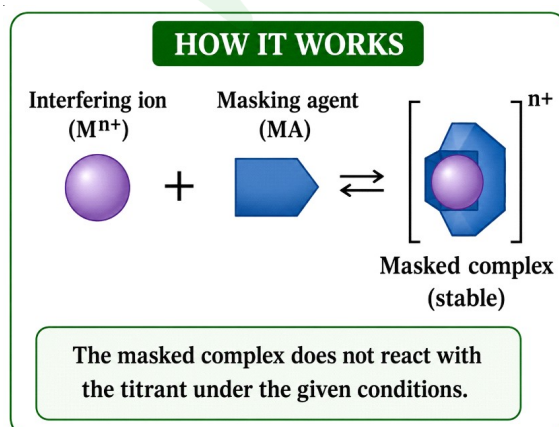


Fig. 3.8: Working of masking agents

Short Answer Questions

1. Write the principle involved in the assay of ammonium hydroxide.
2. Define acid-base titration and state its principle.
3. What is an acid-base indicator? Give two examples.
4. Classify acid-base titrations with suitable examples.
5. What is a non-aqueous titration? Why is it used in pharmaceutical analysis.
6. Classify the solvents used in non-aqueous titrations with one example of each.
7. What is the role of glacial acetic acid in non-aqueous acidimetric titrations.
8. How is perchloric acid standardized for use as a non-aqueous titrant.
9. Why is sodium benzoate assayed by non-aqueous titration.
10. Define precipitation titration and mention its principle.
11. State the principle of Fajans method.
12. What are the main steps involved in gravimetric analysis.
13. Define complexometric titration and mention its principle.
14. What is a metal ion indicator? Give two examples used in EDTA titrations.
15. What are masking and demasking reagents? Give one example of each.
16. Why is pH control important in EDTA titrations.
17. Define oxidation and reduction with one example each.
18. What is a redox titration? Mention its principle.
19. Differentiate between iodimetry and iodometry.
20. Explain the principle, reactions, indicators used, advantages, and applications of permanganometry.

Long Answer Questions

1. Explain the theories of acid-base indicators and discuss the mechanism of colour change during titration.
2. Describe the preparation and standardization of hydrochloric acid and sodium hydroxide solutions with reactions.
3. Discuss the theory of titrations involving strong acids–strong bases, weak acids–strong bases, and weak bases–strong acids with the help of neutralization curves.
4. Describe the preparation and standardization of acidic and basic titrants used in non-aqueous titrations.
5. Explain the estimation of weakly acidic and weakly basic substances by non-aqueous titration. Add the principle and assay of sodium benzoate.
6. Explain the principle, procedure, reactions, advantages, and limitations of Mohr's method, Volhard's method, and Fajans method for precipitation titrations.
7. Explain the gravimetric estimation of barium sulphate, including principle, procedure, reactions involved, and calculations.
8. Classify complexometric titrations and explain the role of metal ion indicators, masking agents, and demasking agents with suitable examples.
9. Explain the assay of Magnesium Sulphate and Calcium Gluconate by complexometric titration, including principle, procedure, reactions, indicators used, and calculations.
10. Discuss the theory of titrations with potassium iodate, including principle, reactions involved, endpoint detection, and pharmaceutical applications.

Gastrointestinal Agents and Radiopharmaceuticals

Unit IV



Syllabus

Gastrointestinal agents

- **Acidifiers:** Sodium acid phosphate and Dilute Hydrochloric acid.
- **Antacids:** Ideal properties of antacids, combinations of antacids, Sodium bicarbonate*, Aluminium hydroxide gel*.
- **Agents promote bowel movements:** Magnesium hydroxide, Sodium orthophosphate, Sodium Potassium tartrate and magnesium trisilicate.
- **Antimicrobials:** Mechanism, classification, Potassium permanganate, Boric acid, Hydrogen peroxide*, Chlorinated lime*, Iodine and its preparations.

Radiopharmaceuticals

- **Basics of radioactivity, applications of radioisotopes of Sodium Iodide I-131, Technetium-99m, Cobalt-60, Phosphorus-32 including safe handling, storage, and disposal of radiopharmaceuticals, adhering to regulatory guidelines for safety.**



GASTROINTESTINAL AGENTS AND RADIOPHARMACEUTICALS



LEARNING OBJECTIVES

After studying this chapter, the learner will be able to:

- To understand the classification, mechanism of action and therapeutic uses of gastrointestinal agents and radiopharmaceuticals.
- To study the ideal properties, preparations and clinical relevance of sodium acid phosphate, dilute hydrochloric acid, sodium bicarbonate, aluminium hydroxide gel, magnesium hydroxide, sodium orthophosphate, sodium potassium tartrate and magnesium trisilicate.
- To explain the pharmacological action of common antacid and laxative compounds such as sodium bicarbonate, aluminium hydroxide gel and magnesium hydroxide.
- To describe the antimicrobial activity and applications of potassium permanganate, boric acid, hydrogen peroxide, chlorinated lime, iodine and iodine preparations.
- To learn the basic principles of radioactivity and the medical applications of sodium iodide I-131, technetium-99m, cobalt-60 and phosphorus-32.
- To understand the safe handling, storage and disposal of radiopharmaceuticals in accordance with radiation safety guidelines.

4.1 GASTROINTESTINAL AGENTS

□ Introduction

- The **Gastrointestinal Tract (GIT)** is a long continuous muscular tube extending from the **mouth to the anus**, responsible for **digestion, absorption and elimination** of food, also known as the **alimentary canal**.
- The GIT consists of the following organs in sequence: **Mouth** → **Oesophagus** → **Stomach** → **Small Intestine** (Duodenum, Jejunum, Ileum) → **Large Intestine** (Caecum, Ascending, Transverse and Descending Colon) → **Rectum** → **Anus**.

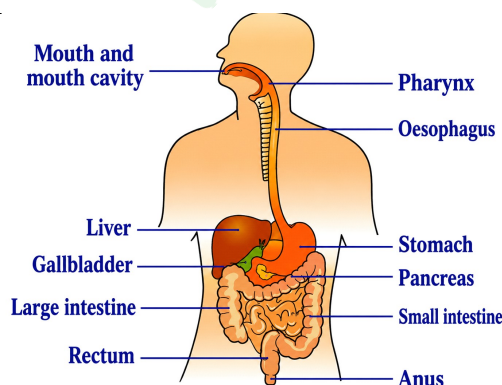


Fig. 4.1: Gastrointestinal Tract

4.1.2 ANTACIDS

- Antacids are drugs that **counteract and neutralize excess gastric HCl**, thereby reducing acidity in the stomach.
- Upon ingestion, they directly react with hydrochloric acid in the stomach and lower the gastric pH to provide relief from **hyperacidity**.
- They are ideally administered 1 to 3 hours after meals and at bedtime, as the presence of food prolongs their neutralizing capacity and extends the buffering time by 1 hour.

❑ Criteria of an Ideal Antacid Preparation

1. Antacids should have a **rapid onset of action** and should exert their effect for a **longer duration**.
2. They should **not be absorbed** systemically to avoid causing **systemic alkalosis**.
3. They should **neither cause constipation nor exert a laxative effect**.
4. They should have a **buffering capacity** in the pH range of **4 to 6**.
5. The reaction between the antacid and HCl should **not produce flatulence**.
6. They should be **finely divided and insoluble in water**.
7. They should **inhibit pepsin** and **not interfere with food absorption**.
8. They should **not cause rebound hyperacidity** or any other side effects.
9. They should be **palatable, inexpensive, stable and readily available**.

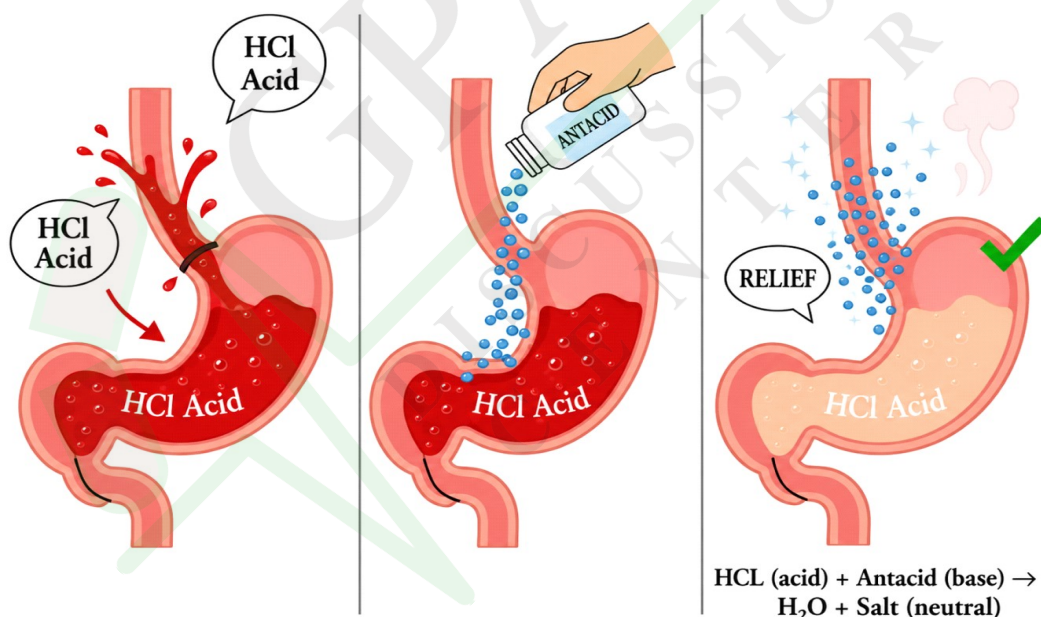


Fig. 4.4: General Mechanism of Action of Antacid

❖ Mechanism of Action of Antacids

1. Chemical Neutralization of Acid

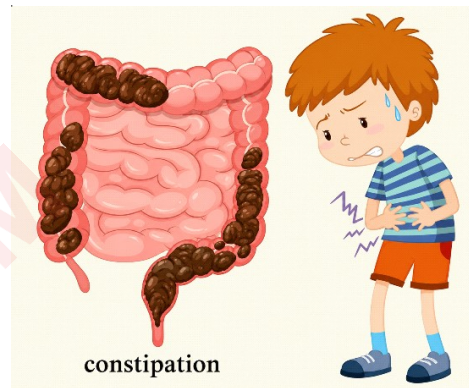
- Antacids **directly neutralize gastric acid**, raising the pH above 4.
- **Examples:** Sodium bicarbonate, calcium carbonate, magnesium carbonate, magnesium oxide.

4.1.3 AGENTS PROMOTE BOWEL MOVEMENTS

❑ Introduction

❖ Cathartics

- Cathartics are drugs that relieve constipation and promote defecation.
- Constipation is a condition characterized by the **delayed passage of faeces** through the intestine. It is associated with difficulty in **defecation and evacuation of the bowel**.
- They are also commonly known as **Laxatives or Purgatives**.
- **Purgatives** are also cathartics, which act similarly but are generally considered as **mild cathartics** in their nature of action that produce semi-fluid or watery evacuation.
- **Laxatives** are also cathartics, which act similarly but are generally considered as **gentle and slow-acting cathartics** that produce **soft and formed stool** without causing complete bowel evacuation.



❖ Types of laxatives

Table 4.2 Types of laxatives

TYPES OF LAXATIVES	DEFINITION
Bulk Forming Laxatives	• They are hydrophilic and indigestible vegetable fibres that act on the intestinal lumen by absorbing and retaining water, thereby increasing faecal bulk and stimulating peristalsis and defecation. Eg: Wheat bran, Ispaghula, Carboxymethyl cellulose, Polycarbophil)
Osmotic Laxatives	• They are non-absorbable and indigestible disaccharides that increase faecal bulk through osmotic and hydrophilic action. Eg: Sorbitol, Lactulose
Lubricant Laxatives	• They are pharmacologically inert mineral oils that soften stool by reducing water absorption from faeces through a lubricating effect. Eg: Liquid paraffin
Surfactant Laxatives	• They are anionic surfactants that act on the intestinal lumen by reducing surface tension of bowel fluids and also act as wetting agents by emulsifying colonic contents, helping water mix into fatty substances of faeces. Eg: Docusate sodium

❖ Types of purgatives

1. Osmotic purgatives - It is of two types:

Table 4.3 Types of Osmotic purgatives

ELECTROLYTE OSMOTIC PURGATIVE	SALINE PURGATIVES
• It contains non-absorbable Polyethylene Glycol (PEG) solution. • Due to its high osmotic nature, it retains water in the intestine. • Ex. Polyethylene Glycol	• These are soluble inorganic salts. • Act by retaining water in the intestine through osmotic effect. • Ex. Magnesium sulphate, Magnesium hydroxide.

2. Irritant purgatives

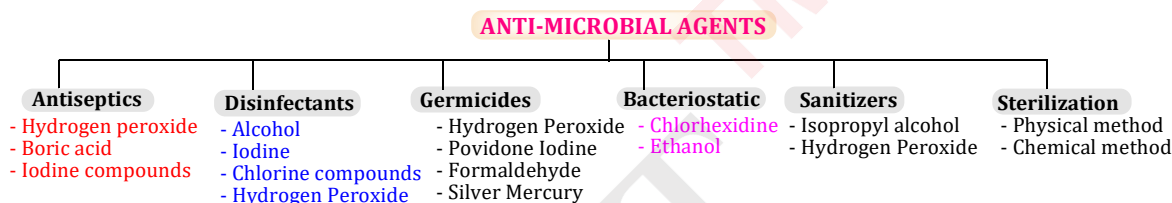
- These are providing semisolid stools. These are further classified into three types:
 1. **Anthraquinone derivatives**- Senna, cascara and aloe.
 2. **Oils**-Castor oil.
 3. **Organic irritants** - Phenolphthalein, bisacodyl, etc.

4.1.4 Antimicrobials

- An antimicrobial is an agent that kills **microorganisms** (microbicide) or stops their growth (**bacteriostatic agent**).
- Antimicrobials – including antibiotics, antivirals, antifungals and antiparasitic – are medicines used to prevent and treat infectious diseases in humans, animals and plants.
- Antimicrobial medicines can be grouped according to the microorganisms they are used to treat.



❑ Classification



❑ Mechanism of Action of Inorganic Antibacterial Agents

❖ General Mechanism

- Ideal antibacterial agents demonstrate **selective toxicity** — targeting prokaryotic microbial cells while leaving host eukaryotic cells unharmed; organic antibacterials like antibiotics achieve this by exploiting key structural differences between the two cell types (e.g., ribosomal subunits, cell wall composition).
- Inorganic antibacterials (e.g., metal ions, nanoparticles) lack this selectivity and act through **non-specific mechanisms**, primarily by indiscriminately denaturing or inactivating proteins in both microbial and host cells.
- Due to this non-specificity, inorganic agents become **cytotoxic at higher concentrations**, limiting their therapeutic use mainly to topical or surface-level applications.
- They act mainly by three mechanisms:
 1. Oxidation
 2. Halogenation
 3. Protein Precipitation

1. Oxidation

- Reducing groups present in most proteins (e.g. sulphhydryl groups, —SH) get oxidized by oxidizing agents.
- Two -SH groups are oxidized to form a disulphide bridge (—S—S—), altering the molecular shape (conformation) of the protein.



- This ultimately leads to the destruction of the protein and the inhibition of biochemical mechanisms.
- **Agents acting by this mechanism:** non-metals, certain anions, hydrogen peroxide, metal peroxides, permanganates, halogens and certain oxo-halogen anions.

2. Halogenation

- **Primary and secondary amide groups** present at peptide linkages in proteins undergo **chlorination**.

- The endpoint is the appearance of a **permanent pale pink color**.
- Since both an acid and an alkali are involved in the reaction, it is termed the **acidimetry-alkalimetry or neutralization method**.

➤ **Procedure:**

- Accurately weigh approximately **2.0 g** of boric acid and dissolve it in a mixture of **50 mL of water** and **100 mL of glycerin** that has been **previously neutralized** with phenolphthalein solution.
- The resulting solution is then titrated against **1M sodium hydroxide (NaOH)** using **phenolphthalein as an indicator** until the endpoint is reached.

❖ **Uses**

- It acts as a weak **bacteriostatic and fungistatic** agent and is used as a local anti-infective in antiseptic creams, lotions, ointments and dusting powders applied to the skin, mucous membranes and eyes.
- Its aqueous solutions are used as **mouthwashes, eye lotions, skin lotions and douches** for bladder and vaginal irrigation, as well as hot fomentations for boils and carbuncles.
- A **1.9% aqueous solution** is iso-osmotic with blood serum, making it suitable for ophthalmic preparations.
- **Boroglycerin glycerite** derived from boric acid is used as a suppository base in pharmaceutical formulations.
- It is used in **buffer systems** such as aluminium acetate solution and ephedrine bitartrate ophthalmic solution.
- It serves as an **insecticide** against cockroaches and black carpet beetles.



❖ **Storage**

Boric acid should be stored in a **well-closed container** to protect it from contamination and moisture.

□ **Hydrogen Peroxide**

- ❖ **Molecular formula:** H_2O_2
- ❖ **Molecular weight:** 34 g/mol
- ❖ **Synonyms:** Perhydroxic acid, O-hydroxyol
- ❖ **Purity Standards:** Contains not less than **5% w/v** and **not more than 7% w/v** of hydrogen peroxide. This corresponds to approximately **20 times its own volume** of available oxygen.
- ❖ **Preparation:**

1. **Industrial Method:**

- Hydrated barium peroxide ($\text{BaO}_2 \cdot 8\text{H}_2\text{O}$) is reacted with sulphuric acid to yield hydrogen peroxide.
- This method is **inefficient**, giving only **10–20% yield**.



2. **Laboratory Method:**

- A thin cream of barium peroxide in water is slowly added to dilute sulphuric acid with constant stirring, **kept in an ice bath** to prevent decomposition.
- The pH is adjusted to acidic, cooled for **1 hour** and the precipitated barium sulphate is filtered off.



➤ Procedure

- To 1 mL of the substance add 20 mL of 1 M sulphuric acid and titrate with 0.02M potassium permanganate.

❖ Uses

- It is a powerful **oxidizing and bleaching agent**, widely used for bleaching hair, fabrics and other materials due to its ability to release nascent oxygen.
- It acts as a mild **antiseptic and disinfectant**, effectively killing pathogenic bacteria such as *E. coli*, *S. aureus* and typhoid bacilli; used in concentrations up to 6% to cleanse wounds and cuts.
- A 1.5% solution is used as a **mouthwash and deodorant gargle** in the treatment of acute stomatitis, while a 3% solution is also commonly used as a mouthwash.
- It is used in dentistry to **clean septic sockets and root canals** and the fine foam of evolving oxygen aids in mechanical cleaning between teeth, crevices and cavities.
- Ear drops containing hydrogen peroxide are used to **remove wax**, dirt and debris from the ear canal.



Fig 4.10: Uses of Hydrogen peroxide

❖ Storage

- Hydrogen peroxide should be stored in a tightly closed, light-resistant glass or plastic container at a temperature not exceeding 30°C.
- Preservative-free solutions must be refrigerated at 2–8°C and should not be stored for prolonged periods.

❑ Chlorinated Lime

❖ **Molecular formula:** $\text{Ca}(\text{OCl})\text{Cl} \cdot \text{H}_2\text{O}$

❖ **Molecular weight:** 144 g/mol

❖ **Synonyms:** Bleaching powder, Chloride of lime, Calcium chloro hypochlorite

❖ **Purity Standards:** Bleaching powder consists of calcium chloro hypochlorite $\text{Ca}(\text{OCl})\text{Cl}$, along with varying amounts of calcium hydroxide and moisture. It contains a minimum of 30% w/w of available chlorine.

❖ Preparation:

- Bleaching powder is prepared by the **action of chlorine gas on slaked lime (calcium hydroxide)**.
- Pure, fairly dried slaked lime is **spread in layers inside lead chambers** and chlorine gas is passed through until the lime is fully saturated. This process takes approximately **12 to 24 hours**.
- Once chlorine absorption is complete, **lime powder is blown into the chamber** to absorb any excess chlorine.

❖ Uses

- Used for **primary diagnosis of intraocular tumours** (eye tumours)
- Also used for **localization of cerebral tumours** (brain tumours)
- Main therapeutic use is the treatment of **Polycythaemia vera** — a condition involving abnormal increase in RBC count
- Also used in the treatment of:
 - ✓ **Myelocytic leukaemia**
 - ✓ **Chronic lymphocytic leukaemia**

❖ Dose

Polycythaemia vera-2 to 10 μCi

Diagnostic purposes-125 to 500 μCi

4.2.3 Handling of Radioactive Materials

- Radioactive materials must **never be touched with bare hands** — always use forceps or suitable instruments.
- **Smoking, eating and drinking are strictly prohibited** in the laboratory where radioactive materials are handled.
- Food contaminated with radioactive material can cause serious damage to internal organs.
- Sufficient **protective clothing and shielding** must be worn at all times while handling radioactive materials.
- **Lead shielding** must be used as lead effectively absorbs harmful radiation.
- Shielding can also be achieved using:
 - ✓ Thick concrete blocks (1 metre thickness) — stop all radiation.
 - ✓ Pure distilled water layer — stops radioactive radiation but allows visible light to pass.



Fig. 4.12: Safety rules for handling of radio active material

4.2.4 Storage of Radioactive Materials

- Radioactive materials must be kept in **properly labelled containers**.
- Containers must be **shielded with lead bricks**.
- Materials should preferably be stored in a **remote corner** of the facility.
- Areas where radioactive materials are **stored or used must be continuously monitored** for radioactivity.
- **Disposal of radioactive materials** must be carried out with extreme care and caution.
- The storage area must be a dry, clean place.
- Storage at **2° to 5°C** will often be required.
- All labelling of pharmaceuticals should clearly show the expiry date.



Unit V

Miscellaneous Compounds



Syllabus

- **Expectorants:** Potassium iodide, Ammonium chloride*.
- **Emetics:** Copper sulphate*, Sodium potassium tartrate.
- **Haematinics:** Ferrous sulphate*, Ferrous gluconate.
- **Poison and Antidote:** Definition, classification of antidotes, Sodium thiosulphate



MISCELLANEOUS COMPOUNDS



LEARNING OBJECTIVES

After studying this chapter, the learner will be able to:

- Understand what expectorants and emetics are, how they work, and learn about commonly used examples such as Potassium Iodide, Ammonium Chloride, Copper Sulphate and Sodium Potassium Tartrate.
- Learn about haematinics, their role in treating anaemia and study the properties and uses of Ferrous Sulphate and Ferrous Gluconate as important iron-containing preparations.
- Understand the meaning of poison and antidote, know the different types of antidotes and learn how Sodium Thiosulphate works as an antidote in poisoning cases.

5.1 EXPECTORANTS

❖ Definition

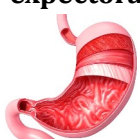
- Expectorants are drugs that help in removing sputum (mucus) from the respiratory tract. They do this two ways — either by **making the mucus thin and watery**, so it is easier to cough out, or by **increasing the amount of fluid in the airways** so that it can be easily removed by the natural sweeping action of cilia and by coughing.
- Based on how they work, expectorants are classified into two types:
 1. **Mucolytic agents** — those that break down and thin the mucus.
 2. **Mucokinetic agents** — those that stimulate the movement and expulsion of mucus from the airways.



❖ Classification of Expectorants

Expectorants are classified into two types based on their mechanism of action:

Table 5.1: Classification of Expectorants

SEDATIVE EXPECTORANTS	STIMULANT EXPECTORANTS
• Sedative expectorants are those that work by stimulating gastric reflexes to produce their expectorant effect. • They are also commonly called stomach irritant expectorants because they irritate the stomach lining to trigger the desired response. 	• Stimulant expectorants are those that work by directly or indirectly stimulating the secretory cells of the respiratory tract. • They increase respiratory secretions in the airways, which in turn reduces the thickness (viscosity) of sputum, making it easier to cough out. 

❖ Uses

1. Used as a **systemic acidifier and diuretic**.
2. Used as an **expectorant and diaphoretic**.
3. Increases respiratory tract secretions and makes mucus **less viscous** — hence used in **cough preparations** along with ammonium carbonate.

❖ Dose

- As a **systemic acidifier**: 1 to 2 gm **four times a day**.
- As an **expectorant**: 0.3 to 0.5 gm.

❖ Storage

- It should be stored in well-closed containers.

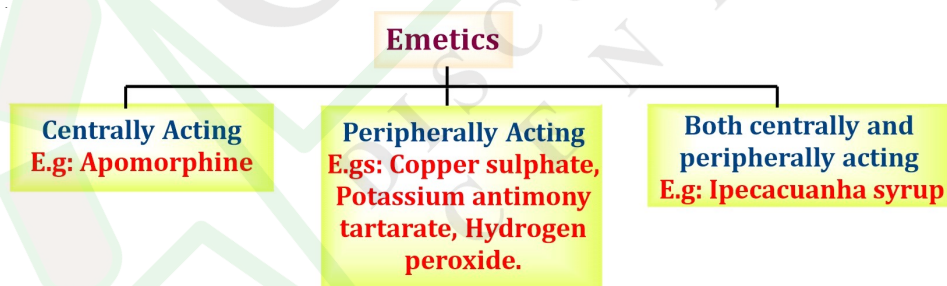
5.2 EMETICS

❖ Definition

- Emetics are drugs that **induce vomiting** either by irritating the gastric mucosa locally or by stimulating the **vomiting centre (VC)** through the **chemoreceptor trigger zone (CTZ)**.
- They are primarily used in the **treatment of poisoning** caused by ingestion of toxic substances.
- They must be administered carefully with proper **therapeutic drug monitoring**.
- Incorrect use of emetics can lead to serious consequences such as: Convulsions, Severe vomiting, Dehydration, Coma, Death.



❖ Classification



❑ Copper Sulphate

- ❖ **Molecular formula**: $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$
- ❖ **Molecular weight**: 249.7 g/mol
- ❖ **Synonyms**: Cupric sulphate
- ❖ **Preparation**



Laboratory Method	• Copper is heated with concentrated sulphuric acid in the presence of air. Without air, the reaction barely occurs. $2\text{Cu} + 2\text{H}_2\text{SO}_4 \longrightarrow 2\text{CuSO}_4 + 2\text{H}_2$ • The solution is then filtered, evaporated and allowed to crystallize to obtain copper sulphate crystals.
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❖ Assay

➤ Principle

- It is assayed by **the cerimetric titration method (redox titration)**.
- The substance is dissolved in water; **dilute sulphuric acid** is added and titrated with **N/10 ceric ammonium sulphate** using **ferroin sulphate** as indicator.
- **End point** is the change of colour from **orange-red to pale green**.
- **Ceric ammonium sulphate oxidizes** ferrous ions to **ferric ions**, and ceric ions are **reduced to cerous ions**.



- Since oxidation and reduction occur simultaneously, it is called **Redox titration**.

➤ Procedure

- Dissolve **0.5 gm of sodium bicarbonate** in a mixture of **70 mL of water** and **30 mL of 1M sulphuric acid**. Wait until **effervescence ceases**.
- Add about **1.0 gm of the substance** (accurately weighed) and **shake gently to dissolve**.
- Titrate with **0.1M ceric ammonium nitrate** using **0.1 mL of ferroin solution** as indicator.
- Continue until the **red colour disappears** (end point).

❖ Uses

- Mainly used as a **haematinic** (increases haemoglobin levels in the blood).
- Also used for the preparation of **ferrous gluconate tablets**.

❖ Dose

- **Prophylactic treatment: 600 mg daily**
- **Therapeutic treatment: 1.2 to 1.8 gm daily** in divided doses

❖ Storage

- Should be stored in **well-closed containers** and protected from **light**.

5.4 POISON AND ANTIDOTE

❑ Introduction

- Antidotes are substances that **specifically react** with an ingested poison, toxic substance or an overdose of a potent drug.
- A **poison** is a substance that causes harmful disturbances in the body, usually through a **chemical reaction at the molecular level**, when a sufficient amount is absorbed.
- Antidotes work by **antagonizing** the effect of the drug, by **neutralizing** the poison, or by **converting** the toxic drug into a **non-toxic metabolite**.

❑ Classification of Antidotes

1. **Chemical antidotes** - The antidote that changes the chemical nature of the poison.

Example- Sodium thiosulphate is a chemical antidote, which converts the systemically toxic cyanide to the non-toxic thiocyanate.

2. **Mechanical antidotes** - The antidote that prevents absorption of the poison into the body.





ASSESS YOURSELF



Multiple Choice Questions

1. Drugs which increase respiratory tract fluid is called

- (a) Expectorant (b) Linctuses
(c) Laxatives (d) Syrups

2. Which is an expectorant

- (a) Sodium Potassium tartrate
(b) Sodium chloride
(c) Potassium iodide
(d) Sodium tartrate

3. The Inorganic compound used as an emetic is

- (a) Iodine (b) Sodium iodide
(c) Copper sulphate (d) Ferric chloride

4. Inorganic saline expectorant is

- (a) Ammonium Chloride (b) Sodium Bicarbonate
(c) Sulphuric Acid (d) None of these

5. Which of the following is NOT a use of Ammonium chloride

- (a) Antihistaminic
(b) Diuretic
(c) Expectorant
(d) Systemic acidifying agent

6. Emetics are used to

- (a) Maintain hydration (b) Induce vomiting
(c) Increase respiration (d) Reduce acidity

7. Which of the following is used as an antidote for cyanide poisoning

- (a) Aluminium hydroxide
(b) Potassium chloride
(c) Zinc sulphate
(d) Sodium thiosulphate

8. Which of the following may be used as chelating agent to treat poisoning by heavy metals such as lead or mercury

- (a) Dimercaprol (b) Sodium phosphate
(c) Bismuth subnitrate (d) None of these

9. Penicillamine is an antidote used in the poisoning of

- (a) Iron (b) Cyanide
(c) Mercury (d) Copper and lead

10. Sodium nitrite ____ type of antidote

- (a) Physical (b) Chemical
(c) Physiological (d) Pharmacological

11. In case of iron poisoning

- (a) Vitamin can effectively treat systemic iron toxicity
(b) Deferoxamine can effectively iron toxicity treat systemic
(c) Edetate calcium disodium can effectively treat systemic iron toxicity
(d) Sodium thiosulphate can effectively treat the systemic iron toxicity

12. Green vitriol is a synonym of

- (a) Ferrous sulphate (b) Magnesium sulphate
(c) Sodium sulphate (d) Calcium sulphate

13. Ferrous sulphate is used to treat

- (a) Acidity (b) Diarrhoea
(c) Vomiting (d) Anaemia

14. Molecular formula of Green vitriol is

- (a) $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (b) $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$
(c) $\text{Fe}_2(\text{SO}_4)_3 \cdot 7\text{H}_2\text{O}$ (d) $\text{Na}_2\text{B}_4\text{O}_7$

15. Which of the following alternatives is a synonym for Ammonium chloride

- (a) Epsom salt (b) Sal ammoniac
(c) Calcium carbonate (d) Sodium chloride

16. Ammonium chloride acts as an expectorant by which mechanism

- (a) Inhibiting mucociliary clearance
(b) Causing reflex stimulation of bronchial glands via gastric irritation
(c) Blocking cholinergic receptors
(d) Direct mucolytic action on disulfide bonds

17. Potassium iodide as an expectorant is particularly useful in

- (a) Dry non-productive cough
(b) Bronchial asthma with thick tenacious secretions
(c) Haemoptysis
(d) Pulmonary fibrosis

18. Ammonium chloride also has which additional pharmacological property besides expectorant

- (a) Antitussive

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A Text Book of PHARMACEUTICAL INORGANIC AND ANALYTICAL CHEMISTRY

As per the New PCI Syllabus NEP 2020 **BP106T**

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