TECHNOCRATS

Lab Work Book of

Pharmaceutical Analysis

(**BP** - 108**P**)

Department of Pharmacy

Lab Manual of **Pharmaceutical Analysis** (BP - 108P)

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Edition:

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Lab Work Book of

Pharmaceutical Analysis (BP - 108P)

(Strictly According to RGPV Syllabus)

/		
Name	:	
Enrollment No.	:	
Institute	:	
Academic Session		

Department of Pharmacy



Vision of the Institute

To grow as an institute of Excellence for Pharmacy Education and Research and to serve the humanity by sowing the seeds of intellectual, cultural, ethical, and humane sensitivities in the students to develop a scientific temper, and to promote professional and technological expertise.

Mission of the Institute

M 1: To inculcate ethical, moral, cultural and professional values in students

M 2: To provide state of art infrastructure facilities to the staff and students so as to enable them to learn latest technological advancements

M 3: State of art learning of professionalism by the faculty and students

M 4: To produce well learned, devoted and proficient pharmacists

M 5: To make the students competent to meet the professional challenges of future

M 6: To develop entrepreneurship qualities and abilities in the students

PROGRAM OUTCOMES (POs)

- Pharmacy Knowledge: Possess knowledge and comprehension of the core and basic knowledge associated with the profession of pharmacy, including biomedical sciences; pharmaceutical sciences; behavioral, social, and administrative pharmacy sciences; and manufacturing practices.
- **2. Planning Abilities:** Demonstrate effective planning abilities including time management, resource management, delegation skills and organizational skills. Develop and implement plans and organize work to meet deadlines.
- **3. Problem analysis:** Utilize the principles of scientific enquiry, thinking analytically, clearly and critically, while solving problems and making decisions during daily practice. Find, analyze, evaluate and apply information systematically and shall make defensible decisions.
- **4. Modern tool usage:** Learn, select, and apply appropriate methods and procedures, resources, and modern pharmacy-related computing tools with an understanding of thelimitations.
- 5. Leadership skills: Understand and consider the human reaction to change, motivationissues, leadership and team-building when planning changes required for fulfillment of practice, professional and societal responsibilities. Assume participatory roles as responsible citizens or leadership roles when appropriate to facilitate improvement in health and well- being.
- **6. Professional Identity:** Understand, analyze and communicate the value of their professional roles in society (e.g. health care professionals, promoters of health, educators, managers, employers, employees).
- **7. Pharmaceutical Ethics:** Honour personal values and apply ethical principles in professional and social contexts. Demonstrate behavior that recognizes cultural and personal variability in values, communication and lifestyles. Use ethical frameworks; apply ethical principles while making decisions and take responsibility for the outcomes associated with the decisions.
- **8. Communication:** Communicate effectively with the pharmacy community and with society at large, such as, being able to comprehend and write effective reports, make effective presentations and documentation, and give and receive clear instructions.
- **9.** The Pharmacist and society: Apply reasoning informed by the contextual knowledge to assess societal, health, safety and legal issues and the consequent responsibilities relevant to the professional pharmacy practice.
- **10. Environment and sustainability:** Understand the impact of the professional pharmacy solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.
- **11. Life-long learning:** Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change. Self-assess and use feedback effectively from others to identify learning needs and to satisfy these needs on an ongoing basis.

PEOs

PEO 1: To inculcate quality pharmacy education and training through innovative Teaching Learning Process.

PEO 2: To promote professionalism, team spirit, social and ethical commitment with effective interpersonal communication skills to boost leadership role assisting improvement in healthcare sector.

PEO 3: To enhance Industry-Institute-Interaction for industry oriented education and research, which will overcome healthcare problems of the society.

PEO 4: To adapt and implement best practices in the profession by enrichment of knowledge and skills in research and critical thinking

PEO 5: To generate potential knowledge pools with interpersonal and collaborative skills to identify, assess and formulate problems and execute the solution in closely related pharmaceutical industries and to nurture striving desire in students for higher education and career growth.

Course Outcomes (COs):

Student will be able to:

- CO1: Develop the Calculations of various standardized solutions.
- CO2: Construct the fundamental methodology to prepare different strength of solutions and can Predict the sources of Errors.
- CO3: Develop Knowledge on Principe and assay procedure of various titrimetric methods.
- CO4: Develop basic knowledge in the principles of electrochemical analytical techniques.
- CO5: Develop interpretation Skills in terms of choice of analytical techniques to perform the Qualitative and Quantitative estimation of different category drugs.

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OBJECT:

To carry out the standardization of 0.1 N HCL using standard solution of Sodium Carbonate.

Apparatus and chemical requirement: Burette, pipette, conical flask, volumetric flask, funnel 0.1 N HC1, Methyl orange Indicator, 0.1 N Na₂CO₃ solution.

THEORY:

It is an example of alkalimetry. When a strong acid titrated with a strong base, the salt produced in the reaction is not hydrolysed and therefore the pH of the resultant solution at the end-point is 7.0.

The following reaction takes place when sodium carbonate is titrated with HC1.

 $Na_2CO_3 + 2HC1 \longrightarrow 2NaC1 + H_2O + CO_2$

In this reaction, for the detection of the end-point methyl orange is used as indicator

PROCEDURE:

- 1. Pipette out exactly 10 ml of 0.1 N Na₂CO₃ solution into a clean conical flask.
- 2. Add 2 or 3 drops of methyl orange indicator.
- 3. Titrate the contents of the flask with 0. N HC1 until red colour is obtained.
- 4. Repeat the titration for concordant values.

N,

Observation:

	Volume of	Burette	e reading	Vol. of Hcl
S.No.	$0.1 \text{ N Na}_2 \text{CO}_3$	Initial	Final	Rundown
	solution (ml)	(ml)	(ml)	(ml)
1.	10			
2.	10			
3.	10			

The normality of Hydrochloric acid is calculated using the formula:

$$\mathbf{V}_1 \mathbf{N}_1 = \mathbf{V}_2 \mathbf{N}_2$$

Where,

 $V_1 = Vol. of N Na_2CO_3 solution$

 $N_1 = Normality of Na_2CO_3 solution = 0.1 N$

 $V_2 =$ Vol. of HCL rundown (Average Burette reading)

$$N_2 = \begin{array}{c} V_1 N_2 \\ V_2 \end{array}$$

Result and Discussion:

Q1.	What are acids.
0.0	
Q2.	What is use of methyl orange.
Q3.	Define strong acid and strong base.
Q4.	What are base.
0.5	What is the use of indicator in titration
Q3.	what is the use of indicator in titration.

OBJECT:

To perform the assay of 20 volume of Hydrogen peroxide solution.

Apparatus and chemical requirement: Conical flask, measuring cylinder, Pipette, burette, Hydrogen Peroxide, Pottasium permagnate, sulfuric acid,

THEORY:

Surfactants are surface active agents which increases the solubility of drug. These moiety have both oil and water loving surfaces which enable the faster solubility and thus dissolution. Solubility is defined in quantitative terms as the concentration of the solute in a saturated solution at a certain temperature and in qualitative terms, it may be defined as the spontaneous interaction of two or more substances to form a homogeneous molecular dispersion. A saturated solution is one in which the solute is in equilibrium with the solvent. The solubility of a drug may be expressed as parts, percentage, molarity, molality, volume fraction, and mole fraction. Due to this major reason Solubility enhancement is one of the important parameters which should be considered in formulation development of orally administered drug with poor aqueous solubility. Solubility is the characteristic physical property referring to the ability of a given substance, the solute, to dissolve in asolvent. Almost More than 90% drugs are orally administered. Drug reproducible bioavailability, pharmacokinetic profile of orally administered drug substances is highly dependent on Solubility of that compound.

PROCEDURE:

- 1. All the apparatus should be washed clearly.
- 2. 1 ml as Hydrogen peroxide acid was taken in a flask.
- 3. 20 ml of H2SO4 was added to the above solution.
- 4. The 0.02M KMNO4 solution was prepared by dissolving 0.3 gm of KMNO4 in 400 ml of water.
- 5. The hydrogen peroxide solution was titrated with KMNO4 solution until colorless.

Observation :

S No	BURETTE READING		
1	INITIAL	FINAL	
2			
3			
4			
5			

RESULT AND DISCUSSION:

Q1.	What is back titration?
Q2.	Difference between qualitative & quantitative analysis?
0.1	
Q3.	What is end point detection?
0 -4	What is analyte?
Q. 1.	what is unaryte.
05.	What is oxidation & reduction?

OBJECT:

To perform the assay of the given sample of ferrous sulphate using standard solution of KMnO₄.

Apparatus and chemical requirement: Burette, pipette, volumetric flask, conical flask, funnel $FeSO_4$, 0.1 N KMnO₄ solution, dil. H₂SO₄, 0.1 N oxalic acid solution.

THEORY:

Ferrous sulphate is an example of hematinic. It is a reducing agent and the titration is an example of Redox titration (Permanganametry). $KMnO_4$ is a powerful oxidizing agent in the presence of dil. H_2SO_4 . During the titration process, ferrous sulphate is oxidised to addition of a drop of $KMnO_4$ produces a permanent pink colour which indicates the end-point. The reactions involved are as follows:

 $2K\underline{MnO_4} + 3H_2SO_4 \longrightarrow K_2SO_4 + 2\underline{MnSO_4} + 3H_2O + 5(O)$ $10FeSO_4 + 5H_2SO_4 + 5(O) \longrightarrow \underline{5Fe_2(SO_4)_3} + 5H_2O$ $2KMnO_4 + \underline{10FeSO_4} + \underline{8H_2SO_4} \longrightarrow K_2SO_4 + 2MnSO_4 + \underline{8H_2O} + \underline{5Fe_2(SO_4)_3} + \underline{3H_2O}$

PROCEDURE:

- 1. Weight accurate 1 g of $FeSo_4$ and dissolve in 20 ml of dil. H_2SO_4 .
- 2. Titrate the contents of the flask with 0.1 N KMnO₄ until a permanent pink colour is obtained.
- 3. Repeat titration for concordant values.
- 4. Enter the value in tabular form.

OBSERVATION:

Titration of Ferrous Sulphate Solution with Standard

KMno₄ Solution

~ > 1	Volume of FeSO	Burette r	Volume of KMnO.	
S.No.	solution (ml) ⁴	Initial (ml)	Final (ml)	rundown (ml)
1	20	-	-	_
2	20	-	-	-
3	20	-	-	-

EQUIVALENT / I.P. FACTOR:

Each ml of 0.1 N KMnO₄ = 0.02789 gm of ferrous sulphate.

CALCULATION:

Percent Purity of FeSO₄

Vol. of $KMnO_4 \times I.P.$ Factor $\times 100 \times N$ of $KMnO_4$ (Actual)

Wt. of $KMnO_4$ in grams $\times N$ of $KMnO_4$ (Expected)

RESULT AND DISCUSSION:

=

Q1.	What do you mean by I.P. factor.			
02.	What do you mean by end-point.			
X				
Q3.	What is Redox titration.			
0.4	What do you mean by Burette reading			
Q - .	what do you mean by Durette reading.			
Q5.	What is the use of $KMnO_4$.			

OBJECT:

To perform the assay of the given sample of sodium bicarbonate.

Apparatus and chemical requirement:

Conical flask, volumetric flask, Burette, pipette, funnel, Sodium bicarbonate, 0.5 N H_2SO_4 , Methyl orange indicator, 0.5 N Na_2CO_3 solution.

THEORY:

Sodium bicarbonate is official in I.P. It is an example of antacid. It is estimated by acidimetric method. The sample is dissolved in water and titrated against standard solution of sulphuric

acid (0.5 N) using methyl orange indicator. The reactions involved are as follows:

 $2NaHCO_3 + H_2SO_4 \longrightarrow Na_2SO_4 + 2H_2O + 2CO_2 \uparrow$

PROCEDURE:

- 1. Weight accurately about 1 gm of sodium bicarbonate sample.
- 2. Dissolve in 50 ml of distilled water.
- 3. Titrate this solution against $0.5 \text{ N H}_2\text{SO}_4$ using methyl orange as indicator.
- 4. Repeat the titration for concordant values.
- 5. Enter the value in tabular form.

OBSERVATION:

Titration of NaHCO₃ with Standard H₂SO₄

		Volume of Sodium	Burette r	Volume	
	S. No.	bicarbonate Solution (ml)	Initial (ml)	Final (ml)	of H ₂ SO ₄ rundown (ml)
	1.	50	0	-	-
	2.	50	0	-	-
	3.	50	0	_	-

EQUIVALENT OR I.P. FACTOR:

Each ml $0.5 \text{ N H}_2\text{SO}_4 = 0.042 \text{ g of NaHCO}_3$.

CALCULATION:

Percentage purity of NaHCO₃

Vol. of H2SO4×I.P. factor×100×N of H2SO4 (Actual)

Wt. of NaHCO3 in grams × N of H2SO4 (Expected)

RESULT AND DISCUSSION:

=

Q1.	What is acid base titration.
Q2.	Give different theory of acid base.
Q3.	What is the role of buffer in pharmaceutical preparation.
Q4.	What are the uses of Sodium bicarbonate.
0.5	
Q5.	what do you mean by normality.

OBJECT:

To Perform the assay of the given sample of Boric acid.

Apparatus and chemical requirement: Test tube, Volumetric flask, conical flask, Burette, pipette, funnel, Boric Acid, Glycerol, Phenolphthalein indicator, 0.1 N Sodium hydroxide solution, 0.1 N oxalic acid solution.

THEORY:

Boric acid is an example of local anti-infective. It is a very weak acid and hence cannot be titrated directly against strong alkali. But on treating with glycerol, glyceryl boric acid is formed which behaves as a strong acid and can be titrated against NaOH using phenolphthalin indicator.

PROCEDURE:

- 1. Weight accurately about 2 gm of boric acid.
- 2. Dissolve in a mixture of 50 ml of water and 100 ml of glycerol (Previously neutralised to solution of phenolphthalein).
- 3. Titrate with 0.1 N NaOH soln. using solution of phenolphthalein as indicator.
- 4. Repeat the titration for concordant values.
- 5. Enter the value in tabular form.

OBSERVATION :

Titration of Boric Acid Solution with Standard NaOH Solution.

	Volume of baric acid	Burette re	Volume of sodium	
S.No.	solution (ml)	Initial (ml)	Final (ml)	Hydroxide solution Rundown (ml)
1.	150			
2.	150			
3.	150			

EQUIVALENT OR I.P. FACTOR:

Each ml of 0.1 N NaOH = 0.06183 g of H₃BO₃

CALCULATION:

Percentage Purity of Boric acid

- $= \frac{\text{Vol. of NaOH} \times \text{I.P. Factor} \times 100 \times \text{N of Na OH (Actual)}}{\text{Vol. of NaOH} \times 100 \times \text{N of Na OH (Actual)}}$
 - Wt. of boric acid in grams \times N of NaOH (Expected)

RESULT AND DISCUSSION:

Q1.	What are the uses of boric acid.
0.1	Circ factors offecting as helitity of descen
Q2.	Give factors affecting solubility of drugs.
Q3.	What is the use of phenolphthalein.
Q4.	What are the importance of solubility in dosage forms.
Q5.	How to enhance the solubility of solid in liquid.

OBJECT:

To Standardize the give solution of 0.1 N KMnO4 using standard solution of oxalic acid.

Apparatus and chemical requirement: Burette, pipette, conical flask, volumetric flask, funnel, 0.1 N KMnO4 solution, dil. H₂SO₄, 0.1 N oxalic acid solution

THEORY:

In volumetric analysis many reactions involve the process of oxidation and reduction. An oxidising agent is estimated by titrating with a reducing agent and vice-versa. These titration are called redox titrations. The Standardisation of KMnO_4 is an example for redox titration. KMnO_4 is a powerful oxidising agent and in acidic medium, it oxidises oxalic acid to CO_2

Procedure:

- 1. Pipette out 10 ml of 0.1 N oxalic acid into a concial flask.
- 2. Add 10 ml of dil H_2SO_4
- 3. Warm the contents of the flask to 70° C.
- 4. Titrate with $KMnO_4$ Solution.
- 5. Continue the titration until a faint pink colour is produced.
- 6. Enter the values in a tabular form.

OBSERVATION:

Titration of 0.1 N Oxalic Acid Solution with KMnO₄ Solution

	Volume of 0.1 oxalic acid solution (ml)	Burette r	eading	Volume of Potassium Permanganate solution
S.No.		Initial (ml)	Final (ml)	
				Rundown (ml)
1.	10			
2.	10			
3.	10			

The normality of KMnO_4 to calculated form

 $V_1 N_1 = V_2 N_2$

Where, V_1 = Volume of 0.1 N oxalic acid soln. = 10 ml

- N_1 = Normality of oxalic acid soln. = 0.1 N
- V_2 = Volume of KMnO₄ (Average burette reading)

 $N_2 = Normality of KMnO_4 = V_1N_1$ $N_2 = V_2$

RESULT AND DISCUSSION:

Q1.	What do you mean by oxidation.
Q2.	What do you mean by reduction.
Q3.	Give classification of solution with example
0.4	Will at our the importance of malow titustion
Q4.	what are the importance of redox titration.
0.5	What is back titration
Q3.	what is back infation.

OBJECT:

To perform the assay of the given sample of Sodium Chloride.

Apparatus and chemical requirement:

Conical flask, volumetric flask, burette, 10 ml pipette, funnel Sodium Chloride, 0.1 N AgNO3 slon, Potassium Chromate solution.

THEORY:

Sodium chloride is an example of electrolyte replenisher. It is assayed by Mohr's method. In this method, the sample is dissolved in water and titrated against a standard solution of AgNO₃ using potassium chromate as indicator. At the end-point it gives brick red colored precipitate due to the formation of Silver Chromate. The reactions involved are as follows:

NaCl	+	AgNO ₃	NaNO ₃	+	AgCl
2 AgNO ₃	+	K ₂ CrO ₄	2KNO ₃	+	Ag ₂ CrO ₄

The method is based on the fact that silver halide is more insoluble than silver chromate. Hence as long as there is any chloride left in the solution, no silver chromate is formed. Even though if formed, will immediately change to Silver Chloride according to the following equation:

 $Ag_2CrO_4 + 2Cl = 2 AgCl + CrO_4^2$

PROCEDURE:

- 1. Take 10 ml of 0.1 N NaCl solutions into a conical flask.
- 2. Add 1 ml of potassium chromate indicator.
- 3. Titrate the contents of the flask against AgNO₃ solution until brick red colored precipitate is formed.
- 4. Repeat the titration for concordant values.
- 5. Enter the values in a tabular form.

OBSERVATION:

Titration of 0.1 N NaCl with Silver Nitrate Solution

	Volume of 0.1	Burette reading		Volume of AgNO ₃
S.No.	N NaCl solution (ml)	Initial (ml)	Final (ml)	solution Rundown (ml)
1.	10	0	-	-
2.	10	0	-	-
3.	10	0	-	-

Calculate the normality of AgNO₃ from the following formula:

 $V_{1}N_{1} = V_{2}N_{2},$ Where, V_{1} = volume of 0.1 N NaCl= 10 ml N_{1} = normality of NaCl =0.1 N V_{2} = volume of AgNO₃ soln. (Average burette reading) N_{2} = Normality of AgNO₃ = ? $V_{1}N_{1}$ N_{2} = V_{2}

For Assay: Weight accurately 0.25 g and dissolve in 50 ml of water and titrate with 0.1 N AgNO₃, using solution of Potassium Chromate as indicator. Repeat the titration for concordant values. Enter the Values in a tabular form.

Titration of Sodium Chloride Solution with Standard AgNO₃ Solution

S.No.	Volume of NaCl	Burette read	Volume of AgNO ₃	
	solution (ml)	Initial (ml)	Final (ml)	solution Rundown (ml)
1.	50	0	-	-
2.	50	0	-	-
3.	50	0	-	-

EQUIVALENT OR I.P. FACTOR:

Each ml of $0.1 \text{ N AgNO}_3 = 0.05845 \text{ gm of NaCl}$.

Percent purity of NaCl

Vol. of $AgNO_3 \times 100 \times N$ of $AgNO_3$ (Actual)

=

Wt. of NaCl in $gm \times N$ of AgNO₃ (Expected)

0.1 N NaCl is prepared by dissolving 2.922 gm in 500 ml of distilled water.

RESULT AND DISCUSSION:

Q1.	What is assay?
Q2.	What is titrate & Titrant?
0.0	
Q3.	What is Normality?
0-4	What are co solvents?
Q	
05.	What are polar and non polar solvents?

OBJECT:

To perform the assay of the given sample of Patassium Cholride KCl.

Apparatus and chemical requirement:

Concial flask, volumetric flask, burette, pipette, funnel, KCl, 0.1 N AgNO₃ Solution, Potassium Chromate solution (5% W/V in H₂O, indicator), 0.1 N NaCl Solution (for the standardization of AgNO₃ solution).

THEORY:

KCl is an example of electrolyte replenished. It is assayed by Mohr's method. In this method the sample solution is titrated with a standard solution of $AgNO_3$ using Potassium chromate indicator. The end-point is indicated by formation of a brick red colored precipitate of Ag_2CrO_4 . The following reactions occur in the assay of KCl.

KCl	+	AgNO ₃	$KNO_3 + Ag$	Cl
K ₂ CrO ₄	+	2AgNO ₃	Ag ₂ CrO ₄	+ 2KNO ₃
			Silver	
			Chromate	

PROCEDURE:

- 1. Weight accurately about 0.15 g of KCl.
- 2. Dissolve in 50 ml of water.
- 3. Add 1 ml of potassium chromate indicator.
- 4. Titrate against 0.1 N AgNO₃ solution until a brick red colored precipitate is obtained.
- 5. Repeat the titration for concordant values.
- 6. Enter the values in a tabular form.

OBSERVATION:

Titration of Potassium Chloride with Standard AgNO₃ Solution.

	Io. Volume of KCl Solution (ml)	Burette read	ing	Volume of AgNO ₃
S.No.		Initial (ml)	Final (ml)	solution Rundown (ml)
1.	50		-	-
2.	50		-	-
3.	50		-	-

EQUIVALENT OR I.P. FACTOR:

Each ml of $0.1 \text{ N} \text{AgNO}_3 = 0.007455 \text{ gm of KCl}$.

CALCULATION:

Percent purity of KCl

Vol. of AgNO₃ × I.P. Factor × 100 × N of AgNO₃ (Actual)

=

Wt. of KCl in $gm \times N$ of AgNO₃ (Expected)

RESULT AND DISCUSSION:

Q.-1. What is end Point? Q.-2. What is back titration ? Q.-3. How to make 0.1 N solution. Q.-4. What is standardization ? Q.-5. How many kind of titration ?

OBJECT:

To Perform the assay of the given sample of $MgSO_4$.

Apparatus and chemical requirement: Burette, pipette, conical flask, volumetric flask, funnel, Magnesium sulphate, 0.05 M EDTA solution, pH buffer solution, Solochrome black T (SBT) indicator, 0.05 M CaCl₂ solution

THEORY:

Magnesium Sulphate is an example of saline cathartic. It is assayed by titration with 0.05 M EDTA using P^{H} 10 buffer and Solochrome Black T indicator. EDTA forms magnesium EDTA complex. The color change of the indicator is form wine red to blue at the end-point.

PROCEDURE:

- 1. Weigh accurately 0.3 g of MgSO_4 .
- 2. Dissolve in 50 ml of H₂O
- 3. Add 10 ml of pH10 buffer solution
- 4. Add 3 or 4 drops of SBT indicator
- 5. Titrate the contents of the flask against M/20 EDTA until the wine red color changes to blue.
- 6. Repeat the titration for concordant values.
- 7. Enter the values in a tabular form.

OBSERVATION

S.No.	Volume of MgSO ₄ Solution (ml)	Burett	e reading	Volume of EDTA solution Rundown (ml)
		Initial (ml)	Final (ml)	
1.	50	0	-	-
2.	50	0	-	-
3.	50	0	-	-

Titration of $MgSO_4$ with Standard EDTA Solution

EQUIVALENT OR I.P. FACTOR:

Each ml of 0.05M EDTA = 0.012319 gm of magnesium sulphate.

CALCULATION: Percent Purity of $MgSO_4$ Vol. of EDTA × I.P. Factor × 100 × M of EDTA (Actual)

=

Wt. of MgS4 in grams \times M of EDTA (Expected)

RESULT AND DISCUSSION:

Q.1	What is solubility?		
0.2	What is Molarity		
Q.2			
Q.3	What solubilizing agent		
Q.4	What is gram equivalent.		
Q.5	What is law of mass action.		

OBJECT:

To prepare and standardize 0.1 N perchloric acid.

Apparatus and chemical requirement:

Burette, pipette, conical flask, volumetric flask, funnel, measuring cylinder, Burette, Perchloric acid, Glacial acetic acid, potassium hydrogen phosphate

THEORY:

Preformulation commences when a newly synthesized drug shows sufficient pharmacological promises in the animal model to warrant evaluation in man. It optimizes the delivery of drug through determination of physicochemical properties of new compound that could effect drug performance and development of an efficacious, stable and safe dosage form. A comprehensive preformulation study helps in understanding the physico-chemical properties of the drug molecule. It provides the foundation for development of a robust dosage form that can sustain the rigors of processing and shelf life. Efforts spent on preformulation provide cost savings in the long run, by reducing challenges during formulation development.

PROCEDURE:

- 1. Weigh 0.7 gm of potassium Hydrogen phosphate.
- 2. 0.1 N HCLO₄ was filled in burette.
- 3. PHP was dissolved in 50 ml Glacial acetic acid.
- 4. Two drops of crystal violet was added to conical flask containing PHP and glacial acetic acid.
- 5. This solution was titrated with $0.1N \text{ HCLO}_4$
- 6. Titration is continued until end point appears giving emerald colour.
- 7. The point at which emerald colour appears is the end point.

FORMULA:

- N1*V1=N2*V2
- N1= Normality of analyte
- V1= Volume of analyte
- N2= Normality of analite
- V2= Volume of Analite

OBSERVATION:

S No	BURETTE READING		
1	INITIAL	FINAL	
2			
3			
4			
5			

RESULT AND DISCUSSION:

Q.-1. What is precipitation Q.-2. Application of Gravimetric titration. Q.-3. What is standardization Q.-4. What is end point Q.-5. Different type of analytical method.

OBJECT:

To determine the concentration of potassium bromide in the given sample by mohr's method.

Apparatus and chemical requirement:

Burette, pipette, conical flask, volumetric flask, funnel, measuring cylinder, silver nitrate, potassium chromate.

THEORY:

The success of a formulation depends on careful selection of the excipients that are added to the

formulation. So careful study of drug and excipient interaction is done. Studies of drug-excipient

compatibility represent an important phase in the preformulation stage of the development of all dosage forms. The potential physical and chemical interactions between drugs and excipients can affect the chemical, physical, therapeutical properties and stability of the dosage form. The present review contains a basic mode of drug degradation, mechanism of drug- excipient interaction like physical, chemical and biopharmaceutical. Different Thermal and Non-thermal method of analysis, Tools and software for incompatibility is also discussed. Once the type of interaction is determined we can take further steps to improve the stability of drug and dosage form. From review, we conclude that consequent use of thermal and non-thermal method provide data for drug- excipient interaction which can further help in selection of excipient for the development of stable dosage form.

PROCEDURE:

- 1. Weigh accurately 0.35 g sample and dissolve in 150 ml of water.
- 2. Titrate with 0.1 M silver nitrate solution using potassium chromate solution as indicator,
- 3. Carry out the calculation with the reading found also calculates the factor.

OBSERVATION:

S No	BURETTE READING		
1	INITIAL	FINAL	
2			
3			
4			
5			

RESULT AND DISCUSSION:

Q.1 What is Morh's method?

_____ Q.2 What is acid base titration? Q.3 What is karl fisher reagent? Q.4 What is law of mass action? Q.5 What is gram equivalent.

REFERENCES:

- [1] Devala Rao G, Practical Pharmaceutical analysis, Second edition 2007, Birla publication Pvt Ltd, New Delhi.
- [2] Beckett AH, Stenlake JB, Practical pharmaceutical Chemistry, Fourth edition 2005, CBS publishers & distributors, New Delhi.
- [3] Vidya sagar G, Basic of drug analysis, first edition 2009, Pharma Med press, New Delhi.